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 Sn_xLi_y When Impacted by a Vertical Column
of Water**

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December 2001

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Abstract

Tin-lithium or Sn_xLi_y , and lead-lithium or Pb_xLi_y , alloys are being considered as liquid breeding materials for nuclear fusion applications. Thus, it is important to understand the safety implications associated with inadvertent contact with water used in an indirect cycle. In an effort to study this interaction, experiments have been conducted with these molten alloys when impacted with a vertical 2.4 m tall column of water at 30°C and 60°C. The qualitative behavior of $\text{Sn}_{75}\text{Li}_{25}$ was compared under similar conditions with other candidate molten metals, specifically tin, lead and a lead-lithium alloy, $\text{Pb}_{83}\text{Li}_{17}$ [7]. Multiple pressure spikes were produced with Sn and Pb, while essentially only one initial pressurization followed by a few strongly damped minor peaks were observed with the different alloys containing lithium. Our current results with tin-lithium are quite similar to tin and lead behavior. Dynamic pressure traces from the physical and chemical reactions are discussed and used to compare the energetics associated with the two different alloys. The pressure traces were used to calculate experimental impulses which represent energetics of the reactions. The impulse ratio of the experimental and the theoretical values remarkably increased in the tests of alloys containing lithium. Hydrogen production from lithium-water reactions was quite rapid and copious. In contrast, hydrogen production with tin-lithium was modest and quite similar to the lead-lithium alloy. It was found that the metal-water interactions of $\text{Pb}_{83}\text{Li}_{17}$ and $\text{Sn}_{75}\text{Li}_{25}$ are quite similar and have significantly reduced energetics from those of pure lithium and other reactive metals.

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Nomenclature

A	area [m ²]
I	impulse [kPa·s·m ²]
g	acceleration of gravity [m/s ²]
H_c	coolant column height [m]
H_{fall}	coolant fall distance [m]
p	pressure [Pa]
p_{inj}	injection pressure [MPa]
T	temperature [°C]
t_{imp}	time for impact [ms]
V	volume [m ³]
W	work [J]
γ	specific heat ratio
ρ	density [kg/m ³]

Subscripts:

c	coolant
$cruc$	crucible
gas	gas, argon
m	molten metal

Superscripts:

$comp$	compression process
$expd$	expansion process
$expt$	experiment
th	theoretical

Chapter 1

Introduction

In designs of nuclear fusion reactors, liquid metals with unique compositions are involved either as a blanket material or a heat transfer medium. Such a metal should have capabilities of breeding and neutron multiplication with a relatively low liquidus temperature; i.e. contain an appropriate amount of lithium as a breeding material. Several different alloy compositions containing lithium have been suggested in this regard. Two such candidate materials are Pb_xLi_y and Sn_xLi_y . The Pb-Li alloy has been proposed for use as a liquid breeder/blanket material in various nuclear fusion applications for over a decade [10] and more recently as a candidate for liquid metal blanket material in the European Union's development for the DEMO reactor [6]. Recently a new candidate metal alloy, Sn-Li, has been proposed for blanket/breeding applications within the ARIES and APEX design studies.

Since these liquid metal alloys are being considered as a coolant, it is important to understand the interactions that might occur if the molten alloy inadvertently contacts water in an accident situation [2, 4, 9]. There are three main areas of concern with the interaction of the alloy with water:

- Possibility of steam explosions that may cause hazardous mechanical energy release and could threaten the vacuum structures and containment.
- Energetic chemical reactions caused by the interaction between water and the lithium component of the alloy. Pure lithium is known to react violently with water. However, the reaction may be substantially reduced when the lithium is

alloyed with another metal such as tin or lead. It is of concern to experimentally measure the extent and rate of the chemical reaction of the specific alloys.

- The associated by-products of the chemical reaction (i.e. hydrogen production and formation of significant quantities of LiOH and LiH). This is a cause of concern due to the presence of the hydrogen and the possibility of hydrogen combustion if it were to mix with air. The presence of the liquidus could cause corrosion and exceed threshold limits for human exposure. The presence of these chemically toxic products may require confinement to prevent public exposure.

Previous investigators [3, 5] have examined some of these issues and have found that there was significant hydrogen production from alloys containing lithium and water. Kranert and Kottowski [5] and later Nelson et al. [7] also observed a dampening of the multiple pressure spikes normally observed when molten lead was forcibly impacted by a column of water. This suppression in the multiple spikes was suggested to be due to the production of hydrogen as a buffer for the pressure oscillations. Recently Anderson et al. [1] compared experimental behavior of the molten $\text{Sn}_{75}\text{Li}_{25}$ alloy to $\text{Pb}_{83}\text{Li}_{17}$ alloy in the aspects of dynamic pressure traces and hydrogen production. Both metal-water interactions were found to be quite similar and were significantly reduced from that of pure lithium and other reactive metals.

Experiments have been performed previously at the University of Wisconsin-Madison to investigate the energetic reaction of Pb-Li [7] and Sn-Li [1] alloy with a water impact generated with a vertical shock tube. The objective of this work is to continue this effort and explore further the hydrodynamic and chemical aspects of different temperatures and composition of molten lithium containing alloys being considered for blanket materials.

Chapter 2

Production of Alloy Sn-Li

The alloys that were tested are $\text{Sn}_{75}\text{Li}_{75}$ and $\text{Sn}_{50}\text{Li}_{50}$. Since these alloys are not commercially available and only small quantities were needed, the samples were prepared by the University of Wisconsin in a manner similar to that discussed by Nelson et al. [8], with some modifications. The entire process of producing the alloy was done in a glove box filled with argon and maintained slightly above atmospheric pressure. Before the beginning of the process, all necessary equipment was installed, including the reactor, tin shot and lithium foil in a sealed glass jar. After measuring a known mass of tin shot into the reactor (Figure 1), the glove box was closed and purged with argon (Figure 2). This was done by flushing the glove box with five times its volume with the argon gas at an oxygen level below 3%. A known mass of lithium strips (approximately 0.8 g each) was slowly added to melted tin shot at a temperature of approximately 500°C (Figures 3 and 4). After the preparation, the alloy was formed into billets using graphite molds. The cooled alloys were weighed, labeled and kept within the inert environment. Core samples of the alloy were obtained and sent to an independent laboratory (Figure 8) for confirmation of the composition (Appendix B).

2.1 Alloy Samples

Two sizes of alloy billets were made. The fixed Li-mass size billet contained 0.83 g of Li. The fixed alloy-volume billet at 12.35 cm³ contained about 1.7 g of Li. These numbers of Li-mass and alloy-volume were calculated from the Pb-Li alloy in the experiments

done by Nelson et al. [7] in order to make the experimental results quantitatively comparable.

The production of the alloy was done three times. The first and second time produced $\text{Sn}_{75}\text{Li}_{25}$ and the last time was to produce $\text{Sn}_{50}\text{Li}_{50}$. The amount of metals that were used in each production was as follows:

1. To make 130 cm^3 of alloy $\text{Sn}_{75}\text{Li}_{25}$ which theoretically equivalents to 933.4 g (density of the alloy was calculated based on mass fraction: Appendix B), the amount of metal needed was

$$\text{Sn} = 915.556 \text{ g Li} = 17.844 \text{ g.}$$

2. To make 8 billets of the fixed Li-mass alloy and 6 billets of the fixed volume alloy of $\text{Sn}_{75}\text{Li}_{25}$, the total mass of the alloy was 879.4 g or 122.47 cm^3 . The amount of metal needed was

$$\text{Sn} = 862.588 \text{ g Li} = 16.812 \text{ g.}$$

3. To make 8 billets of the fixed Li-mass alloy and 8 billets of the fixed volume alloy of $\text{Sn}_{50}\text{Li}_{50}$, the total mass of the alloy was 805.45 g or 116.13 cm^3 . The amount of metal needed was

$$\text{Sn} = 760.960 \text{ g Li} = 44.494 \text{ g.}$$

The amount of each metal in a billet and the measured density are shown in Table

1. The detailed procedure of production of the alloy is explained in Appendix A.

2.2 Observations During the Production Process

1. An exothermic reaction between molten tin and a lithium strip was observed. When the lithium strip was immersed into the molten tin a green flame occurred then changed to a red glowing light (Figure 5 left), followed by some

Parameters	Sn ₇₅ Li ₂₅ Fix volume	Sn ₇₅ Li ₂₅ Fix Li-mass	Sn ₅₀ Li ₅₀ Fix volume	Sn ₅₀ Li ₅₀ Fix Li-mass
Li [g]	1.695	0.83	4.732	0.83
Sn [g]	86.984	42.586	80.925	14.195
Total mass [g]	88.679	43.416	85.656	15.025
Volume [cm ³]	12.35	6.046	12.35	2.166
Density [g/cm ³]	7.180	7.180	6.936	6.936

Table 1: Amount of the metals in each billet at different sizes and compositions.



Figure 1: Tin shot in the reactor.

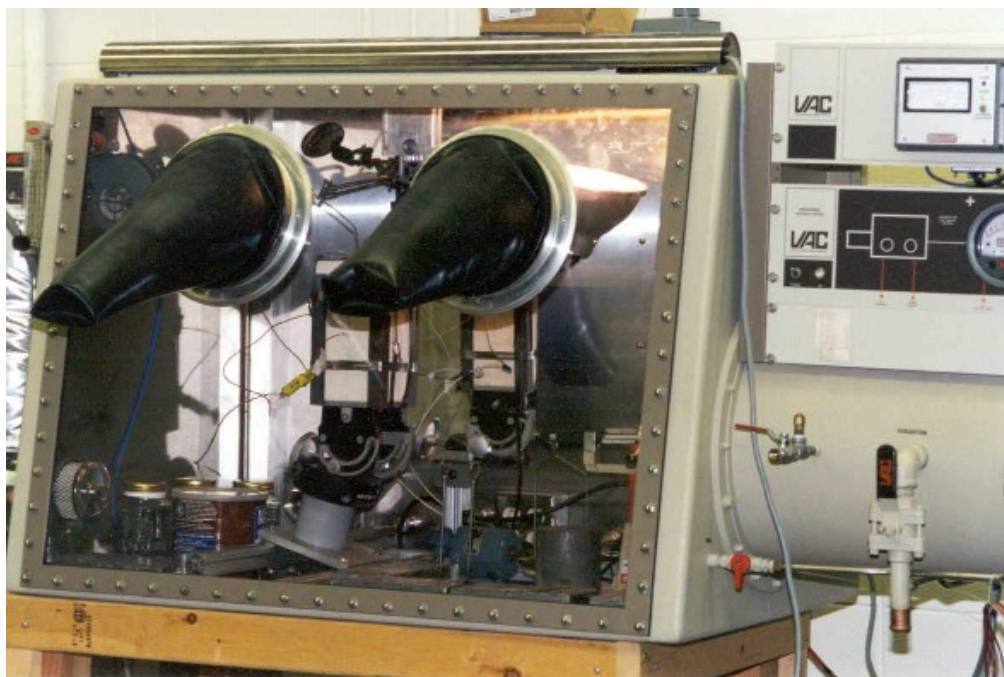


Figure 2: The glove box closed while purging air and flowing argon through.



Figure 3: Li strip on the scale ready to put into the reactor.



Figure 4: A lithium strip was added into the reactor after the tin was melted.

gray smoke. The temperature increased during the reaction and sometimes the molten temperature increased above the heater temperature.

2. After the molten alloy was cast into billets in the graphite mold a crystal structure of the alloy was observed as shown in Figure 6. The crystal structure formed only at the top of the billet.
3. After all the molten alloy was cast, the remaining alloy was allowed to freeze and the reactor allowed to cooled down. This alloy covered the inner part of the reactor and was very difficult to remove (Figure 7) unlike the frozen lead and tin which could be easily removed.

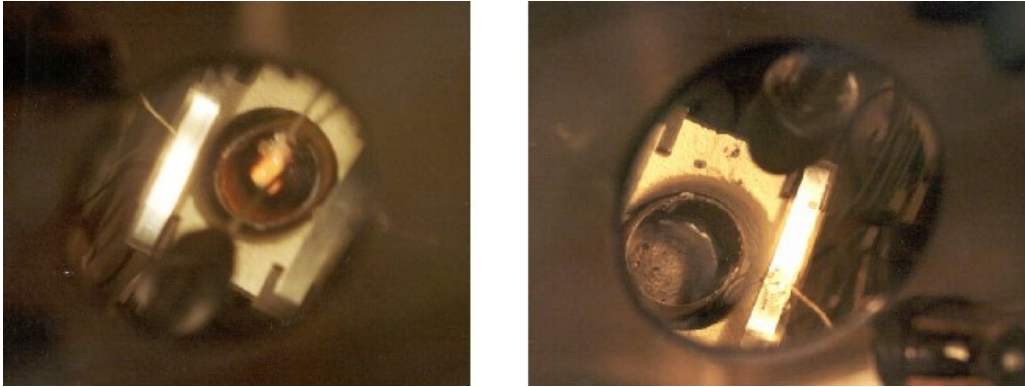


Figure 5: (Left) Li-Sn exothermic reaction, (Right) molten Sn-Li alloy.



Figure 6: Crystal structure was formed at the billet alloy surface.



Figure 7: Reactor and reactor cap after casting the alloy.



Figure 8: Core sample of the alloy to be sent to the laboratory.



Figure 9: Alloy billets were stored in a sealed glass jar under an argon environment.

Chapter 3

Water Impact Experiment

3.1 Liquid Shock Tube Facility

A schematic of the shock tube used in the water liquid metal impact experiments is shown in Figure 10. A shock tube is suitable to study the melt-coolant interaction since it forces direct contact between the melt and the coolant in a one-dimensional characteristic geometry, which simplifies theoretical analysis. The shock tube was designed with the capability to withstand high pressure and temperature peaks due to the combined thermal and chemical reactions associated with these experiments. The tube is comprised of three sections of stainless tubing. The middle section (compression tube) is a 2.54 cm I.D., 3.81 cm O.D. and 198.12 cm long 304 stainless tube. The lower section (reaction tube), 2.40 cm I.D., 3.81 cm O.D. and 17.0 cm long 321 stainless, houses a seamless 316 stainless crucible where the alloy is melted. The upper portion of the shock tube (expansion volume) is a 7.62 cm I.D., 8.89 cm O.D. and 50.8 cm long 304 stainless tube. This portion serves as a gas volume in which argon is introduced to pressurize the water column. A Kapton polymer foil rupture disk (0.5 mm thick) initially separating the compression and the reaction sections of the tube is ruptured at a known pressure causing the water column to accelerate into the liquid metal alloy which sits approximately 42 to 45 cm (depending on the size of the tested alloy) below the rupture disc. The velocity and impact pressure depend on the gas pressure needed to rupture the disc (approximately 1.0 MPa-gage for these experiments). The contact of the molten alloy at an elevated temperature with water

at approximately 30°C to 60°C can lead to an explosive vaporization and chemical reaction.

3.2 Data Acquisition System

A number of pressure transducers (PT) and thermocouples (TC) have been installed at the positions shown in the schematic. A combination of high-speed dynamic pressure transducers, model 112A03 (charge amplifier required) and model 113M213 from PCB Piezotronics, Inc., were used to read dynamic pressure inside the shock tube at various positions of the tube. As soon as the diaphragm ruptured, all the dynamic pressure transducers were synchronized to read the data for 700 ms at 50.0 kHz. Temperature data from the thermocouples was recorded at the initial heating of the test material. Figure 11 shows the data acquisition system used for these measurements. The system comprised of two Windows based computer systems with A/D cards from National Instruments, one digital oscilloscope Hewlett-Packard Infinium and one Hewlett-Packard data logger. All five channels of temperature and a channel for static pressure in the expansion volume were recorded by the data logger system and output to the computer. Four channels of dynamic pressure data were recorded by another computer instrumented with an NI-6110 oscilloscope card. The digital oscilloscope recorded four channels of the dynamic pressure.

3.3 Mass Spectrometer

After each experiment, gas samples from the reaction inside the tube were saved and were analyzed with a mass spectrometer. Figure 12 shows a photograph of the mass spectrometer system. The system consists of three major parts: vacuum chamber, partial pressure gauge and the computer acquisition program.

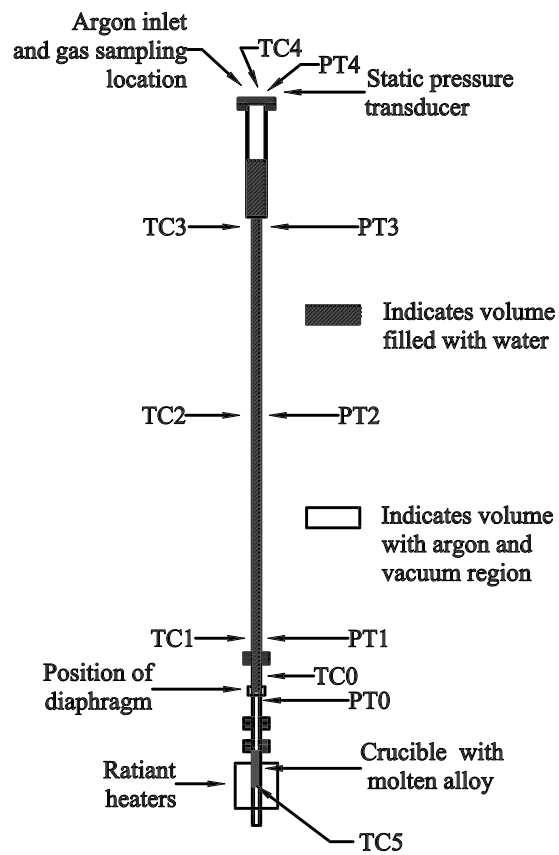


Figure 10: Schematic and photograph of shock tube with locations of pressure (PT) and temperature (TC) probes. At the top is the expansion chamber and at the bottom is the reaction chamber.



Figure 11: Data acquisition system recorded pressure and temperature data from the transducers.

- Vacuum chamber: Contains the gas sample at a very low pressure. The chamber is evacuated continuously by a turbo vacuum pump. The chamber needs to be evacuated for 24 hours after the system is turned on to reach the minimum pressure inside the chamber at approximately 5×10^{-9} torr. Since the vacuum pump has to be running continuously, water coolant is used to remove heat from the pump. The gas sample was analyzed by passing the gas contained in a sampling bottle through a variable leak valve, which maintains a constant flow rate of the gas. An Ion gauge installed in the vacuum chamber was used to measure the total pressure inside the chamber. (This vacuum gauge can measure pressure incorrectly if the partial pressure probe is turned on.)
- Partial pressure gauge: The partial pressure probe is a quadrupole mass spectrometer Balzers QME064. Before taking the measurement, the partial pressure of all detectable gases was recorded to obtain a background measurement, then the gas sample was slowly allowed to enter the vacuum chamber and the leak

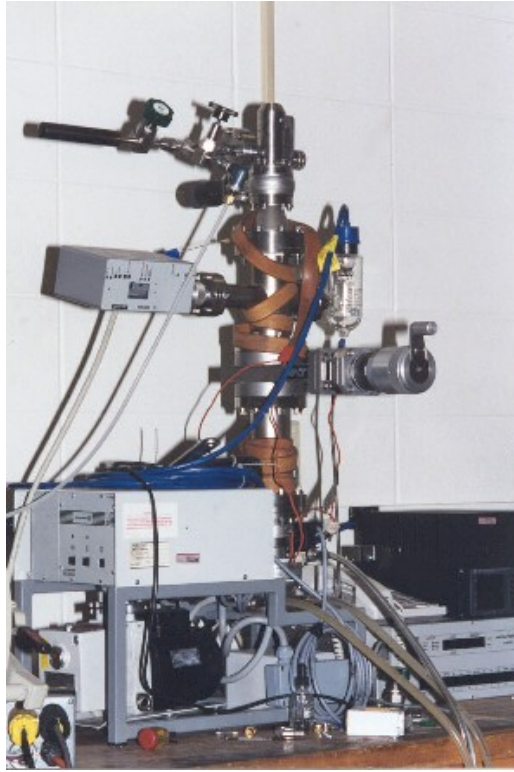


Figure 12: Mass spectrometer system; the partial pressure gauge is installed at the side of the vacuum chamber with the variable leak valve on the top. The acquisition system can be seen at the bottom right of the figure.

flow rate was set to maintain the total pressure constant at 5×10^{-5} torr.

- Acquisition system: The partial pressure data from the gauge was transmitted to the Balzers QMG064 data acquisition system. Outputs were shown and recorded by a PC. 200 data measurements of the pressure data for each sample were recorded at the rate of 1 measurement per second. These data were averaged and recorded in a data sheet.

3.4 Experimental Procedure

Before the experiment, the lower section (without the crucible) of the shock tube needs to be cleaned and dried. The connection between the lower and the middle section had to be greased with vacuum grease to hold and seal the Kapton rupture disc. After the lower and the middle sections were fastened together, crushing the Kapton disc, the coolant water was circulated within the middle section. The crucible with the test material was then installed in the lower section (Figure 13 and 14) while maintaining flow. After the crucible was screwed tightly and sealed with a copper gasket, the argon flow was stopped and evacuation of this section was started. If a leak was observed, it was necessary to reinstall the crucible with a new gasket. This section was low pressure evacuated to about 1 torr of argon gas while melting and heating the sample. Figure 16 shows the reaction chamber while being heated by a radiant heater.

When the desired molten temperature was reached, the lower section pressure was reduced to about 0.3-0.2 torr. Two evacuated gas sample bottles were installed as shown in Figure 15. The circulation of water was ceased and the level was adjusted. This section was then isolated from the circulating pump by high pressure valves. The lower section pressure was then set to 1 torr argon and isolated from the gas flow. At this time the expansion chamber was pressurized with argon until the diaphragm ruptured at approximately 1 MPa. Once the rupture occurred, the shock pressures were recorded with dynamic pressure transducers.

After a few minutes the radiant heaters used to melt and heat the alloy were switched off. Gas samples were taken immediately (30 s) and after 5 minutes. After shock rupture, these samples were then analyzed on the mass spectrometer for hydrogen content in order to give an idea of the extent of the reaction between the lithium and water. After cooling (approximately 4-10 hours) the water from the shock tube was drained and kept for lithium hydroxide titration analysis which gave a second measurement of the extent of the lithium/water reaction. The remnants of the molten



Figure 13: The test material is put into the crucible in the glovebox then seal the crucible with a rubber cap.

alloy, which was essentially fragmented, were collected and weighed and saved for further analysis. Figures 17 and 18 show collection of the test material and contaminated water after the experiment. A detailed procedure of the water impact experiment can be found in Appendix C.

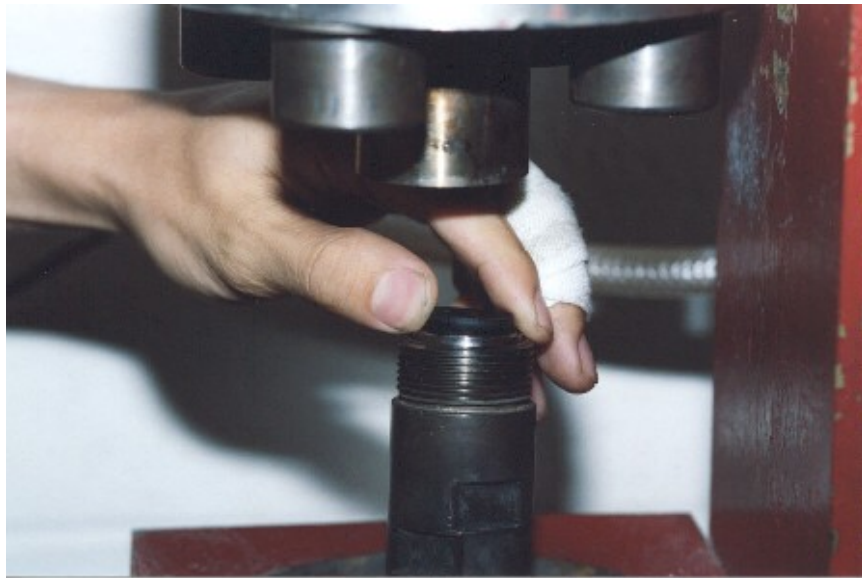


Figure 14: The crucible and the test material in a metal housing are being installed in the lower section of the shock tube. The rubber cap is removed under flowing argon.



Figure 15: Two gas sample bottles are installed on the top of the expansion chamber.

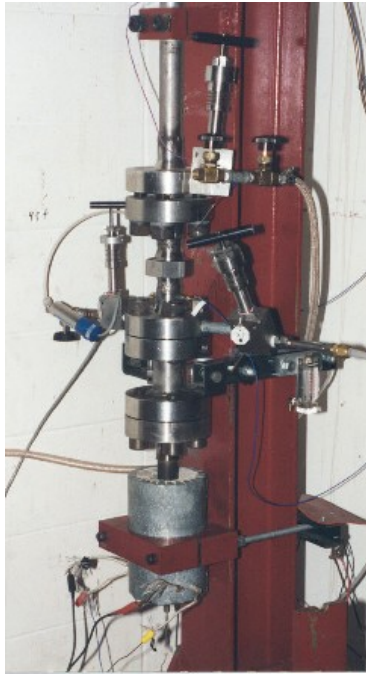


Figure 16: The crucible with the test material in the metal housing is being heated.



Figure 17: The test material and contaminated water were saved after each experiment for further analysis.



Figure 18: Some of the alloy debris can not be removed from the copper gasket.

Chapter 4

Experimental Results and Discussion

In this section we present a series of experimental results with the Sn-Li alloy. Some of these experiments will then be compared to past results obtained by Nelson et al. [7] for the Pb-Li alloy. Table 2 presents the experimental matrix. The first three experiments were base case studies to ensure consistency between our experiments, Expt#2, 3 and 4, and those of Nelson et al. [7] and Vukovic [11]. Note that Nelson's tests with lead-lithium are also included in Table 2. The first was a water impact with an empty crucible at room temperature (Expt#2), the second was a water impact with an empty heated crucible (Expt#3) and the third was a water impact into pure tin at approximately 580°C (Expt#4). Expt#8 and #9 were experiments using a fixed volume of the alloy at 12.35 cm³, which is the same volume as the Pb₈₃Li₁₇ test alloy by Nelson et al. The remainder of the experiments were water impacts into fixed Li-mass Sn-Li alloy at various temperatures of the molten metal and coolant. Expt#19 was an experiment with an alloy of different composition, namely Sn₅₀Li₅₀, but Li-mass was still fixed. Experiments identified B-25, B-31, B-35, B-50 and B-52 were the experiments with Pb₈₃Li₁₇ performed by Nelson et al. [7]. These test results will be compared to the current work in order to study differences in behavior between the two eutectic alloys.

4.1 Dynamic Pressure Traces

In the case of the empty cold crucible (Figure 19) the dynamic pressure trace from PT0 shows a series of several pressure spikes indicative of a damped water hammer oscillation, where the peak pressure decreased with each impact. Figure 20 shows pressure trace PT0 in the case of a heated empty crucible. Similar behavior of the oscillations have been observed except at a lower frequency for the heated case due to vapor explosion when water contacted the hot crucible. In these figures the present data are plotted along with that of Nelson et al. [7] to show the consistency in the results. These initial scoping tests are also shown to be consistent with those of Vukovic's [11], Figure 22. There is a slightly different oscillation time perhaps due to the different driving pressures (Table 2). In the case of pure tin there was a significant reduction in the number of oscillations. The initial peak pressure pulse was similar; however, the second pressure spike occurred at a later time, 150 ms, as compared to the 75 ms of the empty crucible and had a much higher pressure than the first peak. This observation was consistent with that observed by Nelson et al. and Vukovic and is most likely due to vapor generation damping the initial oscillations. The dynamic pressure plots of the pure lead taken from Nelson et al. and pure tin are plotted in Figure 21. The comparison between the two is again quite similar. Figure 23 shows the pressure trace in the test of pure lead by Vukovic [11]. The trace shows a very good agreement with the current study and Nelson et al.

Figure 24 shows PT0 data (refer to Figure 10) of the $\text{Sn}_{75}\text{Li}_{25}$ along with Nelson et al.'s PT0 data of the $\text{Pb}_{83}\text{Li}_{17}$. Figure 25 also shows PT0 data of two alloys but at a higher temperature of coolant. These four cases all have the same amount of Li (0.83 g). All curves remarkably exhibit the same trend. These results were not comparable to Vukovic's since studies of lithium or lithium alloy were not present in her experiments.

Expt#7 (Figures 26 and 27) will be the base case to study the effect of temperature

Expt#	Sample	Description	P-inj [psig]	T-cruc [°C]	T-coolant [°C]
2	-none-	Empty cold crucible	126.76	26.38	28.49
B-25	-none-	Empty cold crucible by Nelson et al. [7]	144.80	14.60	27.30
3	-none-	Empty hot crucible	119.47	587.01	30.62
B-31	-none-	Empty hot crucible by Nelson et al. [7]	138.00	576.70	30.70
4	Pure Sn	12.35 cm ³ of pure Sn	117.00	579.00	28.58
B-35	Pure Pb	12.35 cm ³ of pure Pb by Nelson et al. [7]	147.90	590.20	29.10
7	Sn ₇₅ Li ₂₅	fix Li-mole	143.47	593.79	27.60
B-50	Pb ₈₃ Li ₁₇	same mole of Li by Nelson et al. [7]	153.10	598.50	27.40
8	Sn ₇₅ Li ₂₅	fix alloy volume	79.96	597.83	27.68
9	Sn ₇₅ Li ₂₅	fix alloy volume	120.39	599.96	26.91
14	Sn ₇₅ Li ₂₅	fix Li-mole, hot coolant	123.31	649.90	62.96
B-52	Pb ₈₃ Li ₁₇	same mole of Li by Nelson et al. [7]	132.30	594.10	59.60
15	Sn ₇₅ Li ₂₅	fix Li-mole, high molten	128.73	812.24	29.17
16	Sn ₇₅ Li ₂₅	fix Li-mole, hot coolant high molten	133.40	804.71	63.82
19	Sn ₅₀ Li ₅₀	fix Li-mole	131.94	608.73	27.83

Table 2: Experimental matrix

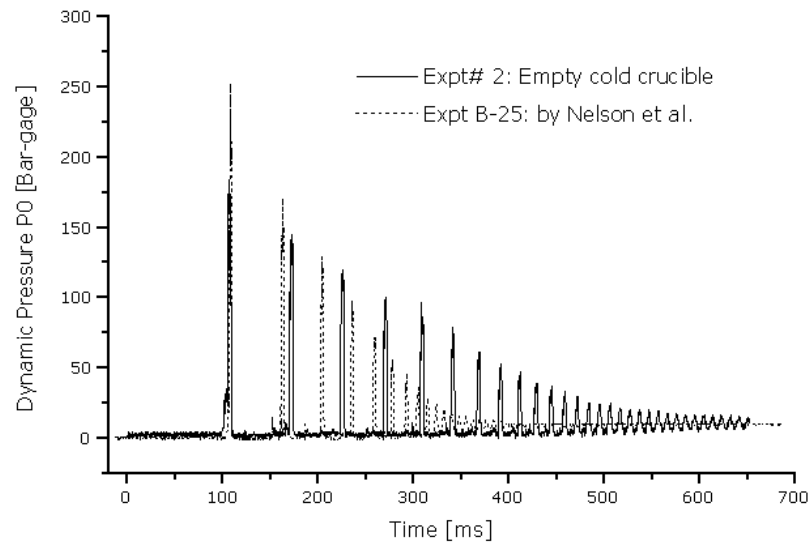


Figure 19: PT0 dynamic pressure trace for cold crucible tests.

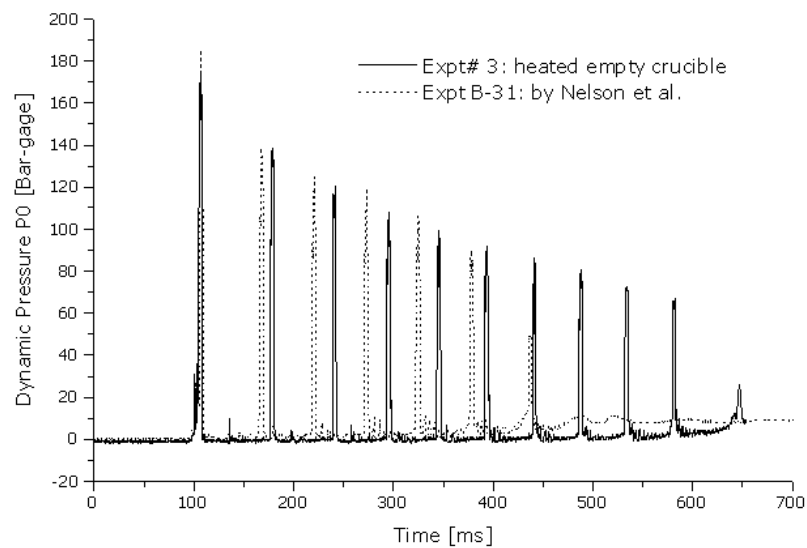


Figure 20: PT0 dynamic pressure trace for heated crucible tests.

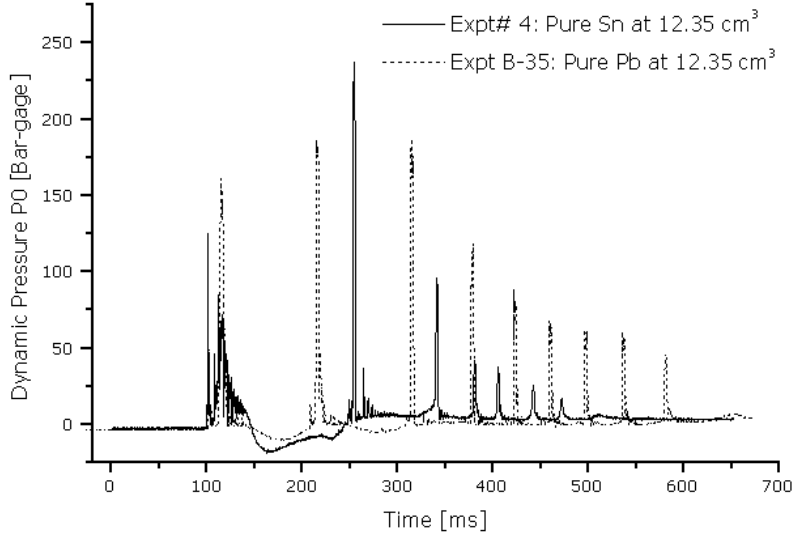


Figure 21: PT0 dynamic pressure trace for pure tin and pure lead [7] with the same volume, 12.35 cm^3 .

and composition on the pressure trace, impulse and hydrogen production. In the case of higher temperature of molten alloy at 800°C in Expt#15, PT0 is very similar to the base case as shown in Figure 28 and slightly different in PT4 (Figure 29). The second positive peak of PT4 in Expt#15 is higher than in Expt#7; this is perhaps due to more hydrogen and/or steam production. Expt#16 was conducted with a higher molten temperature and coolant temperature. PT0 is again similar to the base case, as shown in Figure 30, except for slightly higher pressure of the first peak, approximately 170 bar-gage compared to 160 bar-gage in Expt#7. The second peak occurs at approximately the same time (~ 170 ms after the first impact) as other tests with $\text{Sn}_{75}\text{Li}_{25}$ fix Li-mass alloy. A large difference of the pressure trace shows up in PT4 with a very high positive peak right after the impact. The first positive peak of PT4 in Figure 31 raises up to approximately 3.4 bar-gage which is about 3 times higher than that of the base case. The second positive peak then significantly decreases to

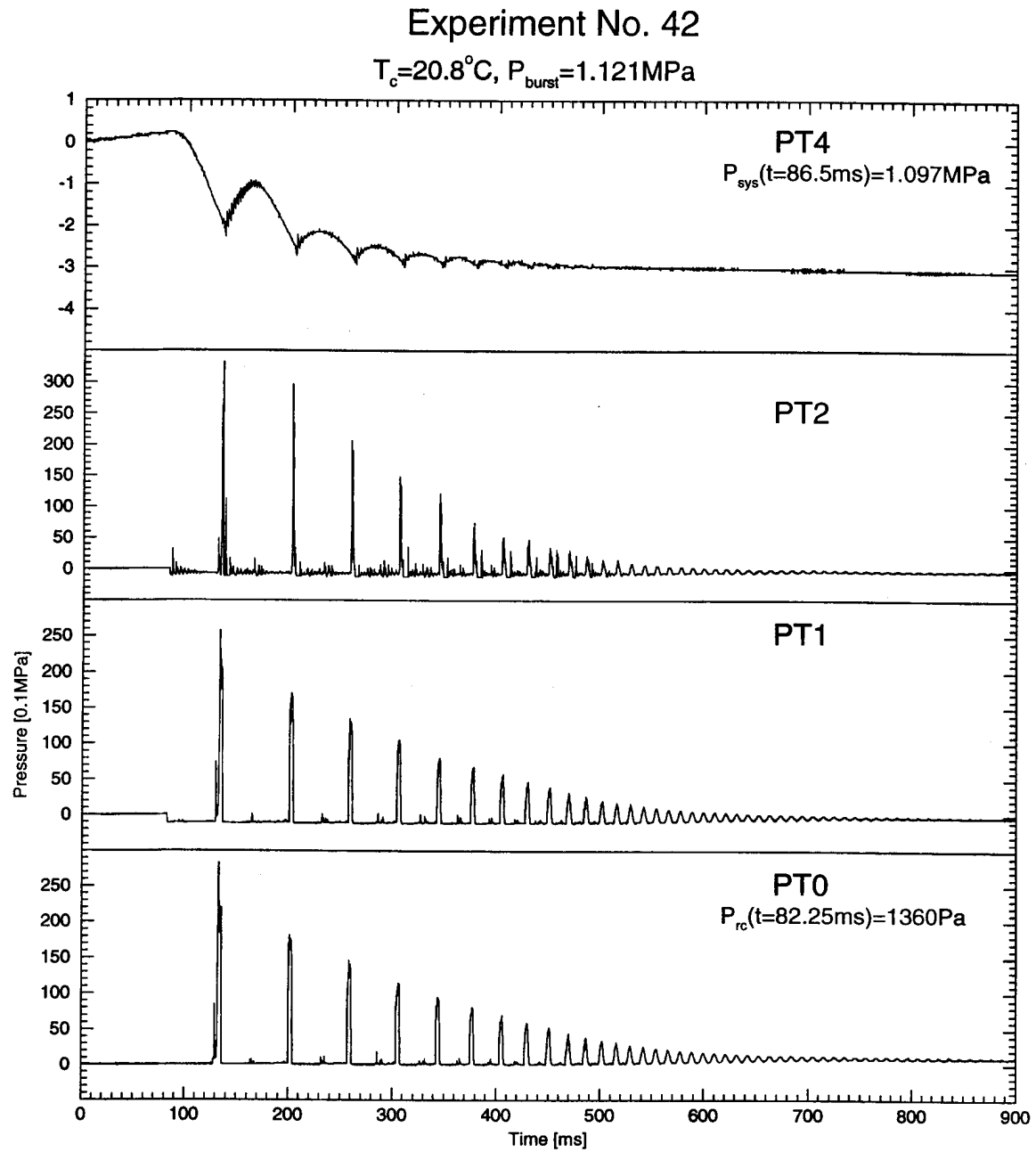


Figure 22: Dynamic pressure trace for unheated empty crucible test from Vukovic [11].

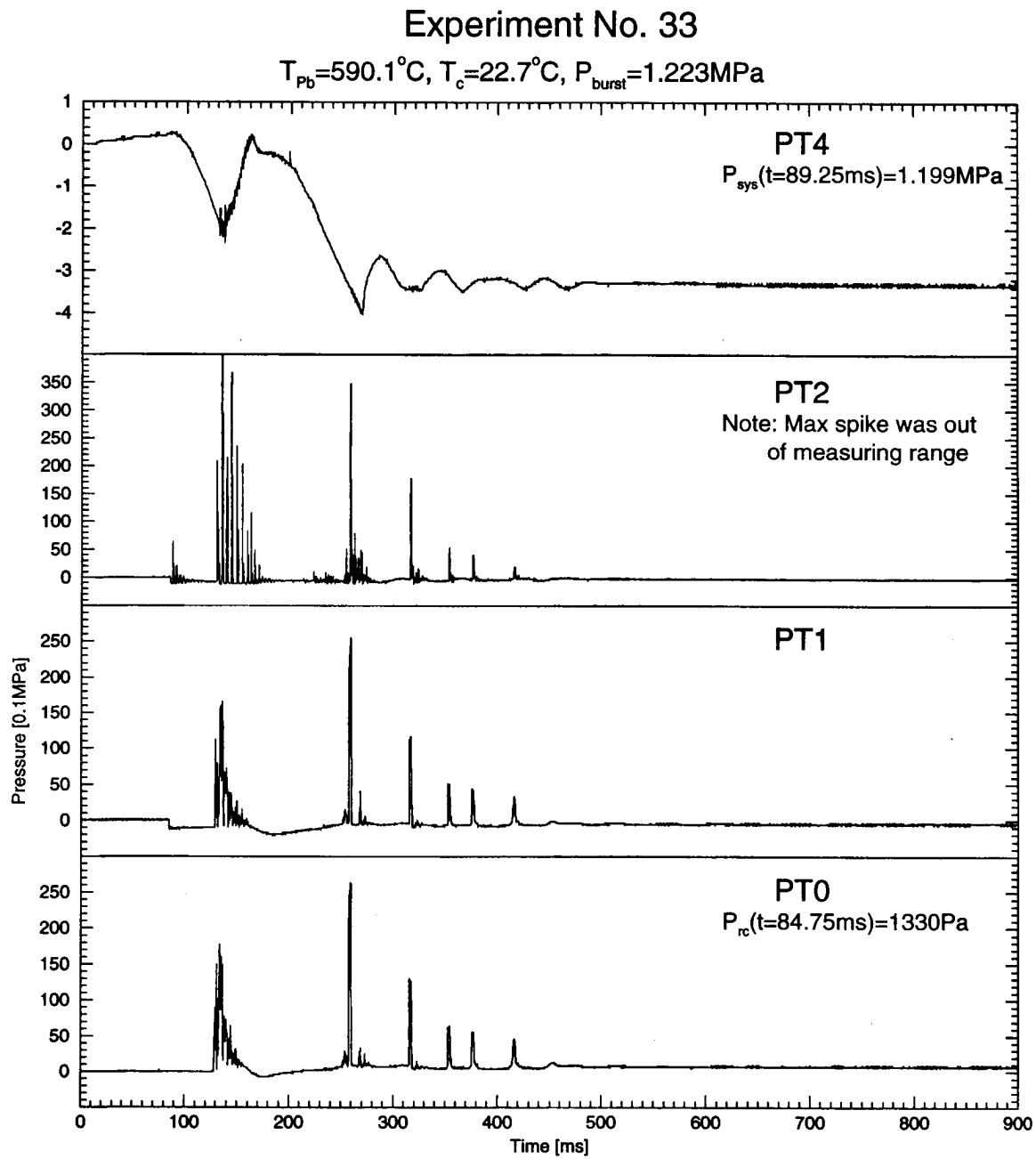


Figure 23: Dynamic pressure trace for pure lead from Vukovic [11].

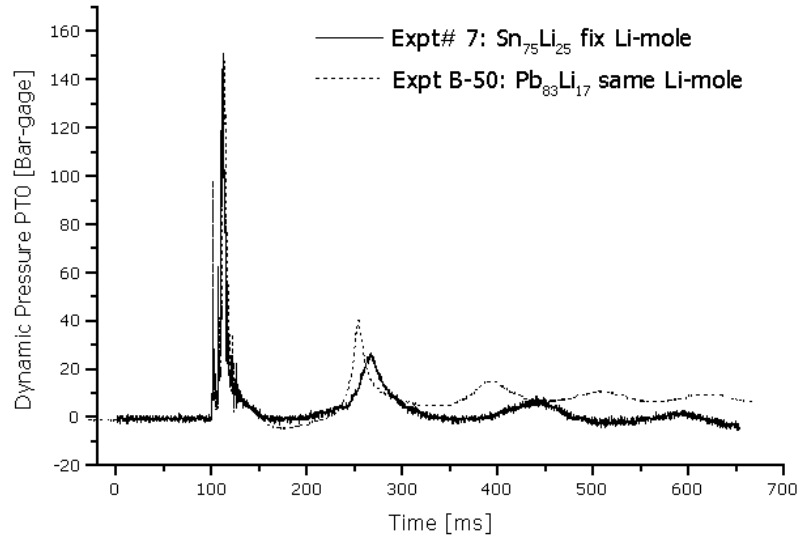


Figure 24: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ and $\text{Pb}_{83}\text{Li}_{17}$ with same amount of Li, 0.83 g.

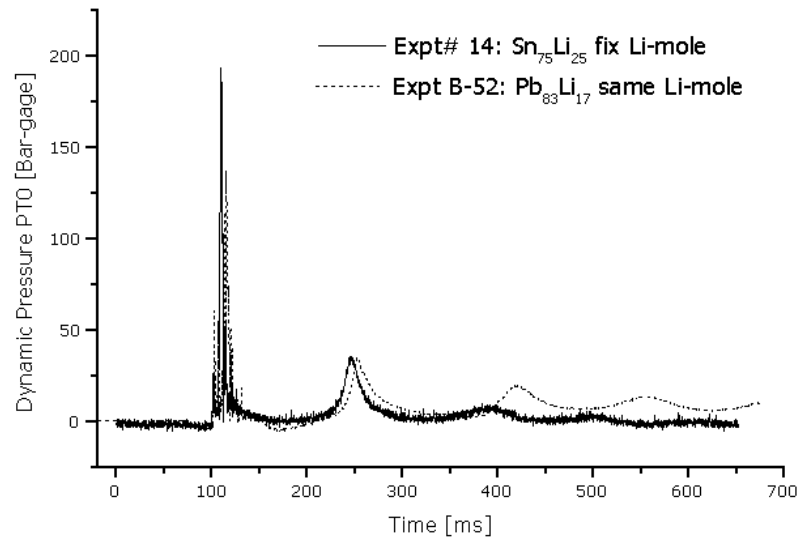


Figure 25: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ and $\text{Pb}_{83}\text{Li}_{17}$ with same amount of Li, 0.83 g, at higher temperature of coolant.

the same order as in Expt#15. A higher production rate of steam has been expected to cause this event since the higher temperature of water coolant can be vaporized more easily, especially when contacted with the higher temperature molten material in the reaction chamber.

In the experiment of $\text{Sn}_{50}\text{Li}_{50}$ alloy at the base temperature in Expt#19, no significant difference to the base case in PT0 was observed (Figure 32). PT4 in Figure 33 shows a higher positive first peak right after the impact. This is obviously due to more hydrogen production from the higher ratio of lithium-to-tin present in this alloy. All the experiments discussed above were testing of the Sn-Li alloy at same amount of lithium, 0.83 g. Expt#8 and #9 were conducted with not a fixed mass but a fixed volume (12.35 cm^3 of $\text{Sn}_{75}\text{Li}_{25}$). In these tests there was more lithium which resulted in an increasing hydrogen production from the reaction. Figure 34 shows PT0 of Expt#9. The trace is similar to the base case except a slightly longer delay in the occurrence of the second peak was observed. This is because with a greater amount of molten alloy in the reaction chamber, the reaction products (steam and hydrogen) spend more time oscillating along the tube. PT4 as shown in Figure 35 has a higher first positive peak which one could expect is due to greater hydrogen production. Note that a noticeable small spike before the first major spike in PT0 of every experiment is expected, caused by the shock wave below the diaphragm that propagates to the reaction chamber due to the water column impact when the diaphragm ruptures.

4.2 Impulses

The experimental impulse could be calculated from the dynamic pressure trace of PT0 by integrating the pressure with respect to the sampling time numerically from the start of the pressure until the completion of the impulse, which can be observed from the corresponding highest peak of PT4. For example, to calculate the first impulse of

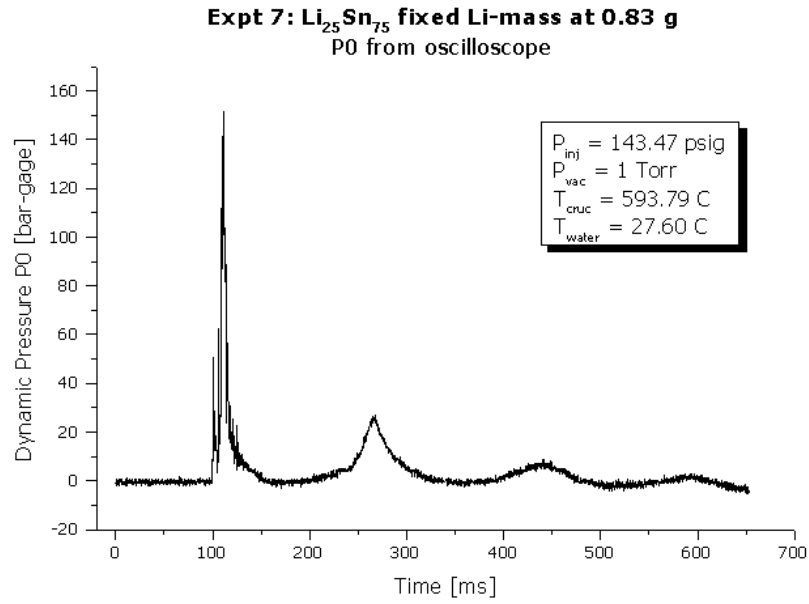


Figure 26: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at base temperature of molten alloy.

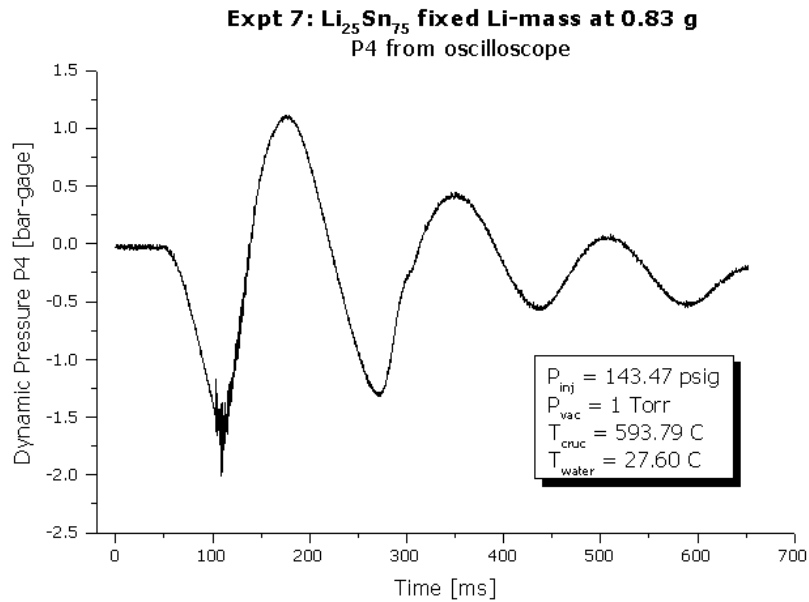


Figure 27: PT4 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at base temperature of molten alloy.

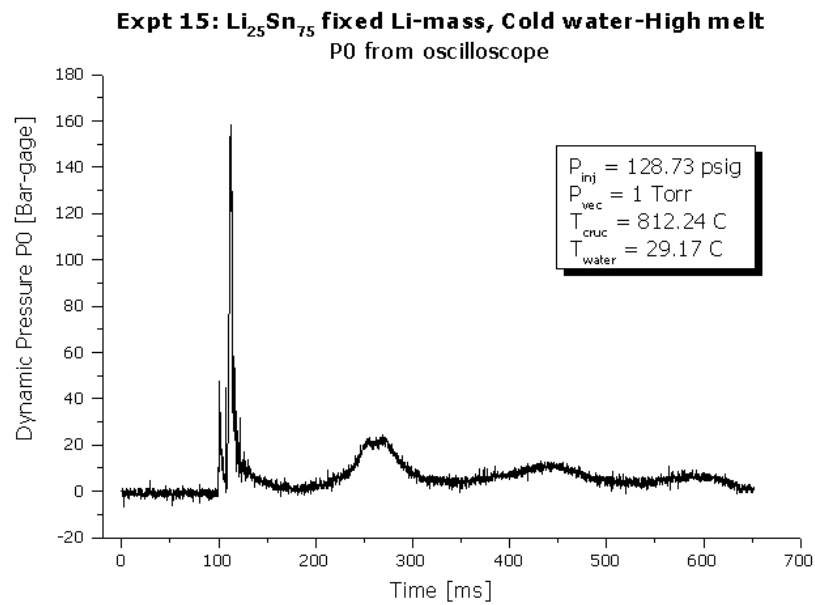


Figure 28: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at higher temperature of molten alloy.

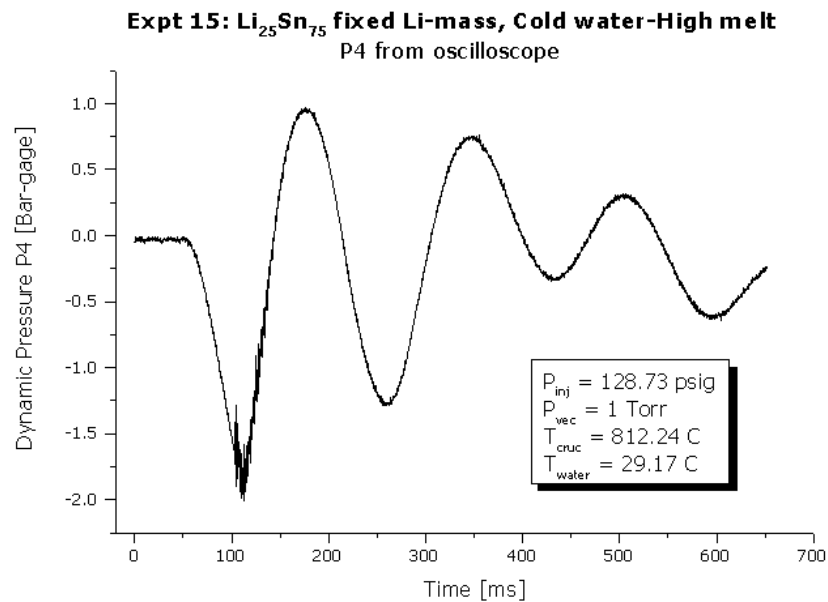


Figure 29: PT4 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at higher temperature of molten alloy.

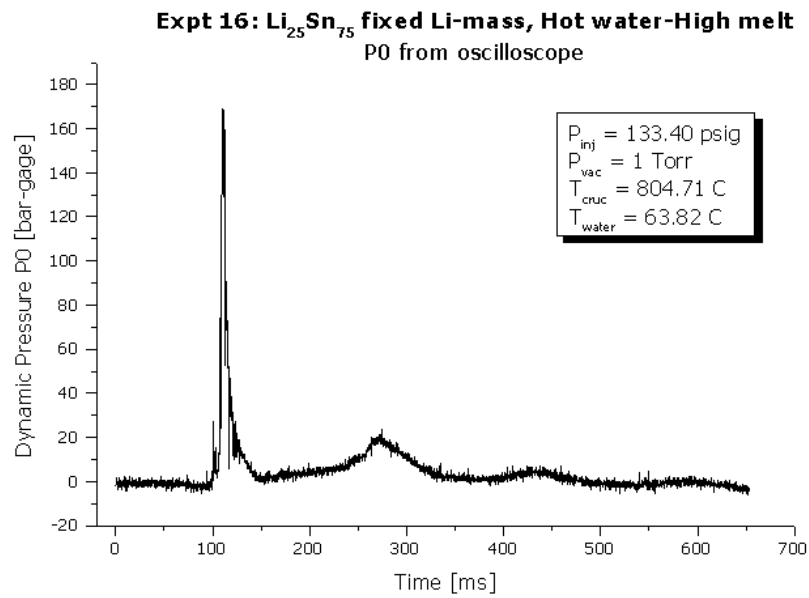


Figure 30: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at higher temperature of both coolant and molten alloy.

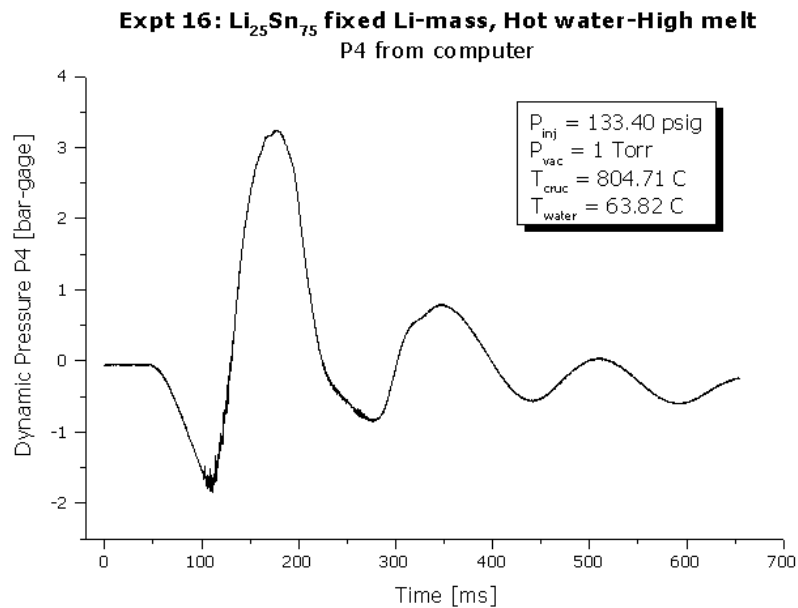


Figure 31: PT4 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ at higher temperature of both coolant and molten alloy.

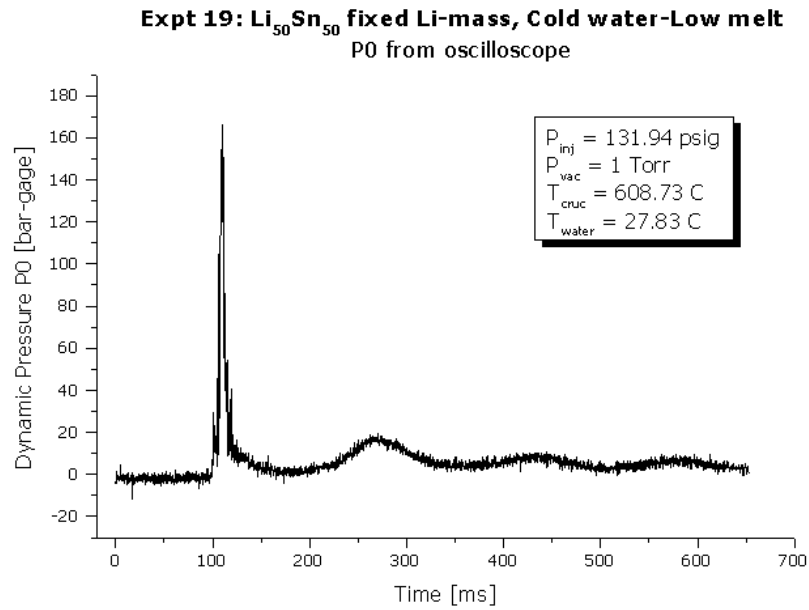


Figure 32: PT0 dynamic pressure trace for $\text{Sn}_{50}\text{Li}_{50}$ at base temperature of both coolant and molten alloy. Amount of lithium in the alloy is fixed at 0.83 g.

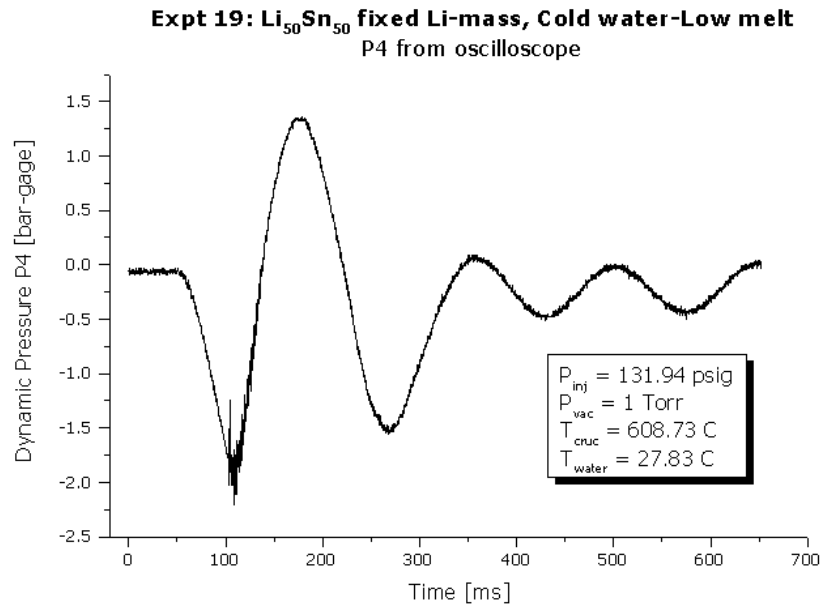


Figure 33: PT4 dynamic pressure trace for $\text{Sn}_{50}\text{Li}_{50}$ at base temperature of both coolant and molten alloy. Amount of lithium in the alloy is fixed at 0.83 g.

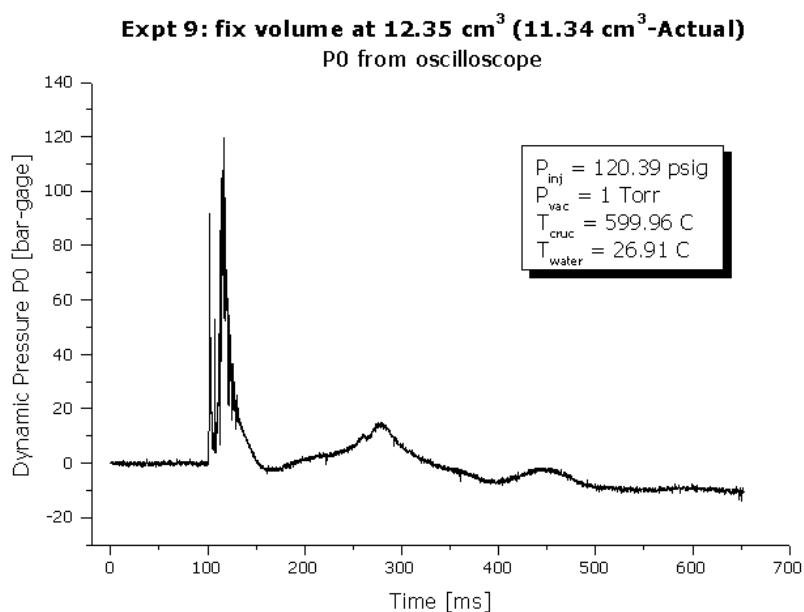


Figure 34: PT0 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ fixed volume at 12.35 cm³.

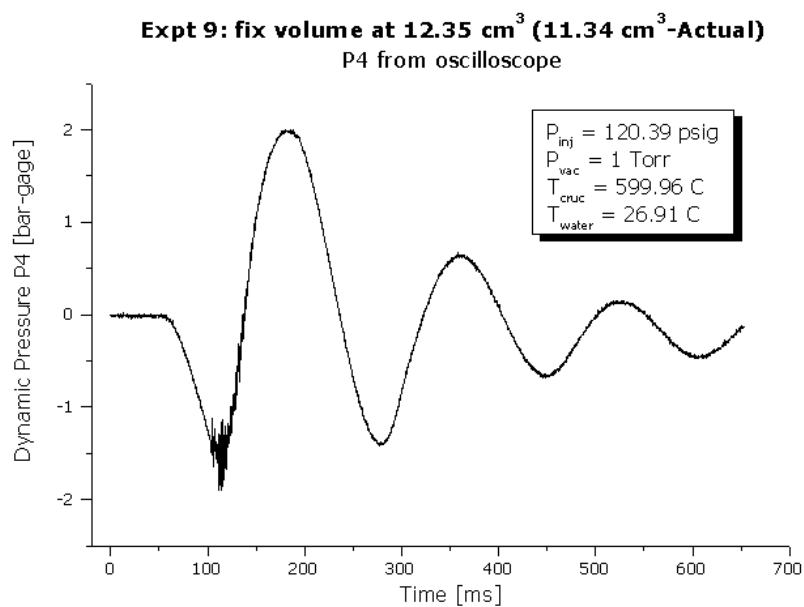


Figure 35: PT4 dynamic pressure trace for $\text{Sn}_{75}\text{Li}_{25}$ fixed volume at 12.35 cm³.

Expt#7, start from the first pressure point of PT0 at time 100 ms (Figure 26), then integrate numerically with the sampling time, 0.02 ms, until the time that the highest point of the first positive peak of PT4 has been reached (Figure 27). In this case, PT0 has been integrated from time 100 ms to 175 ms. The second and third impulse were also calculated from PT0 using a similar criterion. The integration starts from the point where the pressure begins to rise for the next peak of PT0 then integrating until the highest point of the next positive peak of PT4.

The theoretical impulse value was calculated according to the equation

$$\left(\frac{I}{A}\right)^{th} = 2(p_{inj} + \rho_c H_c g) t_{imp}^{th} \quad (1)$$

from [11], where the theoretical time for impact, t_{imp}^{th} , was calculated from

$$t_{imp}^{th} = \sqrt{\frac{2H_{fall}\rho_c H_c}{p_{inj} + \rho_c H_c g}} \quad (2)$$

and $H_{fall} = 2.63$ m for our experiments. Note that only hydrodynamic behavior of the coolant was taken in to account to calculate the theoretical impulse. Theoretical and experimental values of the impulse per unit area are shown in Table 3. Figure 36 is a plot of impulse ratio of the first measurement to the theoretical value, I_1^{expt}/I^{th} . It can be clearly seen from the plot that all of the non-reactive impacts, i.e. Expt#2, 3, 4, B-25 and B-31, give an impulse ratio less than 1.0 or in other words the experiment impulses are less than the theoretical impulse. This is because several lost factors of the impact such as friction, heat transfer, vibration and even sound were not taken into account to calculate the theoretical impulse. Impulse ratios of all the reactive impacts, the experiments with Li -alloy, are all greater than unity. These can clearly represent the effect of an energetic reaction of the impacts since reaction of water and lithium in the test alloy yields an explosive production of hydrogen. At the base temperature of coolant and molten metal, Sn₇₅Li₂₅ in Expt#7 yields the same order of impulse as Pb₈₃Li₁₇ in Expt#B-50. As one would expect, the higher temperature

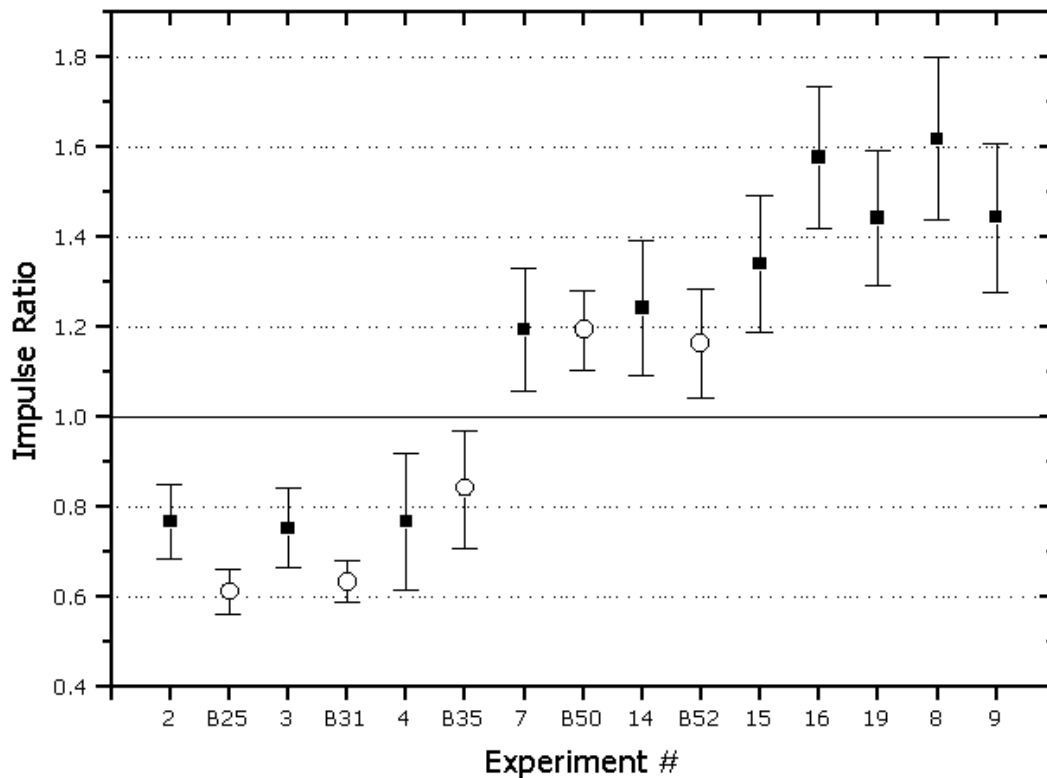


Figure 36: Impulse ratio of the first pulse, I_1^{expt}/I^{th} .

of either coolant or molten alloy, the higher the resulting impulse (as seen from the plot of Expt#14, 15 and 16). Impulse from Expt#19 is higher than from Expt#7 at the base conditions since the higher ratio of lithium-to-tin in $\text{Sn}_{50}\text{Li}_{50}$ yields a stronger explosive reaction. Expt#8 and 9 were expected to yield higher impulse because of the increased amount of lithium which could react.

4.3 Work Ratio

The ratio of compression work to expansion work of the gas expansion vessel can be used as another representation of the energy generated from the reaction. The higher

Expt#	Sample	H_{fall}	t_{imp}^{th}	$(\frac{I}{A})^{th}$	$(\frac{I}{A})_1^{expt}$	$\frac{I_1^{expt}}{I^{th}}$	$(\frac{I}{A})_2^{expt}$	$\frac{I_{(1+2)}^{expt}}{I^{th}}$
2	-none-	0.4554	51.597	92.851	71.280	0.768	46.640	1.278
B-25	-none-	0.4554	48.362	99.061	60.720	0.613	42.400	1.047
3	-none-	0.4554	53.101	90.221	67.904	0.753	48.227	1.296
B-31	-none-	0.4554	49.509	96.767	61.280	0.633	42.760	1.082
4	Pure Sn	0.4254	51.845	86.320	66.246	0.767	86.250	1.776
B-35	Pure Pb	0.4254	46.262	96.737	81.084	0.838	85.450	1.730
7	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , base T_m	0.4404	47.773	96.979	115.777	1.194	111.811	2.359
B-50	Pb ₈₃ Li ₁₇ base T_c , base T_m	0.4254	45.488	98.381	117.134	1.191	118.090	2.403
8	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	0.4254	62.268	71.870	116.329	1.619	24.240	1.978
9	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	0.4254	51.132	87.523	126.300	1.443	72.916	2.293
14	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , base T_m	0.4404	51.424	90.094	111.927	1.242	116.531	2.550
B-52	Pb ₈₃ Li ₁₇ high T_c , base T_m	0.4254	48.842	91.626	106.700	1.165	118.880	2.475
15	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , high T_m	0.4404	50.361	91.996	123.172	1.339	162.343	3.118
16	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , high T_m	0.4404	49.496	93.603	147.616	1.577	158.681	3.289
19	Sn ₅₀ Li ₅₀ fix Li-mole base T_c , base T_m	0.4504	50.324	94.155	135.833	1.443	173.099	3.296

Table 3: Theoretical and experimental impulse per unit area in each experiment

the work ratio the more energetic the reaction upon impact.

The work done during gas expansion or compression from state 1 to state 2 was calculated using the following equation

$$W_{1\rightarrow 2} = \frac{p_2 V_2 - p_1 V_1}{1 - \gamma} \pm m_{gas} g H_{fall} \quad (3)$$

where p and V are gas pressure and volume in the expansion vessel respectively, and the specific heat ratio $\gamma = 1.667$. Gas expansion and compression were assumed to be isentropic processes. Sign “+” corresponds to expansion, while “−” corresponds to gas compression. To calculate the first expansion work due to the rupture $W_{1\rightarrow 2}$, the injection pressure, p_{inj} , would be counted as p_1 and the first minimum pressure PT4, which is where the water column hits the crucible, as p_2 . The volume V_2 was simply calculated from the initial gas vessel volume plus the volume that the water column traveled. The first compression work $W_{2\rightarrow 3}$ was calculated by taking the maximum pressure of the first positive peak of PT4 as p_3 and the volume V_3 was calculated from an isentropic relation of the compression process. Calculations of work for secondary bounces would be done in a similar manner.

The expansion and compression work in each bounce of each experiment are shown in Table 4. Note that the positive work indicates work done *by* the system (gas in the expansion vessel) and negative work indicates work done *on* the system. Since there are no records of PT4 of Expt#8, B-25, B-31 and B-35, an analysis of the work ratio could not be included. Table 5 shows quantitative values of the work ratio and Figure 37 shows them in graphical form. Since we have observed that the reaction occurs mostly during the first impact, only the work ratio of the first impact would yield a reasonable representation of the energetics of the reaction. From Figure 37, the work ratio of the empty crucible tests are very small since there were no reactions. Experiments conducted with pure tin yield a significantly higher work ratio due to the vapor explosion. Work ratios of the Sn-Li experiments are even larger due to the reaction of the water with lithium.

Expt#	Sample	W_1^{expd}	W_1^{comp}	W_2^{expd}	W_2^{comp}	W_3^{expd}	W_3^{comp}
2	-none-	337.91	-68.03	58.72	-54.32	35.94	-35.06
3	-none-	334.36	-72.24	49.12	-62.09	39.95	-35.65
4	Pure Sn	325.74	-219.97	219.97	-107.18	66.31	-78.73
7	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , base T_m	314.74	-260.68	197.10	-144.15	81.08	-55.43
B-50	Pb ₈₃ Li ₁₇ base T_c , base T_m	380.75	-180.20	323.60	-143.50	108.57	-46.78
9	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	345.61	-317.64	263.49	-162.51	101.13	-66.66
14	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , base T_m	335.97	-270.42	172.68	-243.42	269.19	-0.09
B-52	Pb ₈₃ Li ₁₇ high T_c , base T_m	336.24	-187.09	244.23	-463.53	144.30	-9.72
15	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , high T_m	353.55	-249.40	173.85	-162.58	86.17	-56.36
16	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , high T_m	325.34	-407.57	300.88	-127.44	102.22	-49.01
19	Sn ₅₀ Li ₅₀ fix Li-mole base T_c , base T_m	369.45	-283.87	220.36	-137.06	48.06	-36.25

Table 4: Expansion and compression works

Expt#	Sample	$(W^{comp}/W^{expd})_1$	$(W^{comp}/W^{expd})_2$	$(W^{comp}/W^{expd})_3$
2	-none-	0.201	0.925	0.975
3	-none-	0.216	1.264	0.892
4	Pure Sn	0.675	0.487	1.187
7	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , base T_m	0.828	0.731	0.684
B-50	Pb ₈₃ Li ₁₇ base T_c , base T_m	0.473	0.443	0.431
9	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	0.919	0.617	0.659
14	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , base T_m	0.805	1.410	0.0
B-52	Pb ₈₃ Li ₁₇ high T_c , base T_m	0.556	1.898	0.067
15	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , high T_m	0.705	0.935	0.654
16	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , high T_m	1.253	0.424	0.480
19	Sn ₅₀ Li ₅₀ fix Li-mole base T_c , base T_m	0.768	0.622	0.754

Table 5: Compression to expansion work ratios

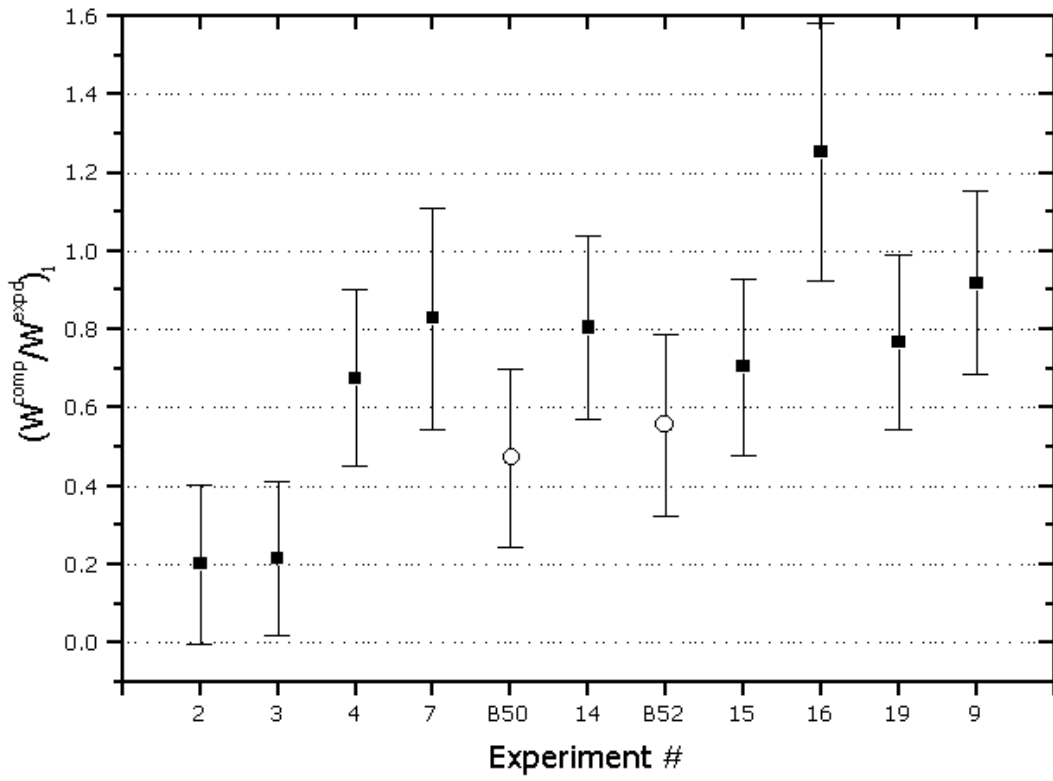
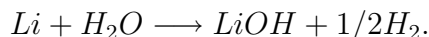


Figure 37: Work ratio of the first pulse, $(W^{comp}/W^{exp})_1$.

4.4 Hydrogen Production

Approximately 30 seconds after the impact, the first gas sample was taken by opening the multi-way valve to the evacuated gas sample bottle “A”. Four minutes later (five minutes after the impact), the second gas sample was taken with bottle “B”. These two gas samples were analyzed immediately for composition by the mass spectrometer. The results were in percent partial pressure, then calculated to mmole using the ideal gas law applied to the gas vessel. Detailed measurements from the mass spectrometer are shown in Appendix E. The uncertainties of the measurements from the mass spectrometer are on the order of 14%.

After letting the system cool down for about 4-10 hours, water from the experiment was collected for composition analysis by the University of Wisconsin Soil Plant Analysis Lab. Concentrations of LiOH in the water in the reactive experiments were determined by titration methods, then used to calculate the number of moles of hydrogen that could be produced according to the chemical reaction



The uncertainty of the titration method measurements was approximately 10%. The amount of hydrogen produced measured from the two methods is shown in Table 6. Figure 38 is a plot of hydrogen measurement from the mass spectrometer at five minutes after the impact.

From Figure 38, the amount of hydrogen produced becomes greater at a higher temperature of either coolant or molten alloy. Reaction of $Sn_{50}Li_{50}$ in Expt#19 yields the most hydrogen among the same Li-mole tests. Hydrogen produced from Expt#8 and 9 were expected to be higher because of the greater amount of lithium in the alloys. The amount of hydrogen produced can be used to calculate the amount of lithium reacted in the test materials using the chemical balance. Table 7 presents the percent of lithium-reacted necessary to produce the amount of hydrogen from the three

Expt#	Sample	Alloy mass [g]	H ₂ – Mass Spec ($\pm 14\%$)		H ₂ – Titration ($\pm 10\%$)	
			<i>mmole</i>	$\frac{mmole}{g-alloy}$	<i>mmole</i>	$\frac{mmole}{g-alloy}$
7	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , base T_m	42.14	26	0.63	37	0.88
B-50	Pb ₈₃ Li ₁₇ base T_c , base T_m	116.59	33.7	0.289	42.9	0.368
14	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , base T_m	42.82	17	0.39	36	0.86
B-52	Pb ₈₃ Li ₁₇ high T_c , base T_m	122.42	34.4	0.281	45.5	0.372
15	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , high T_m	41.47	23	0.56	40	0.98
16	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , high T_m	40.00	27	0.67	27	0.68
19	Sn ₅₀ Li ₅₀ fix Li-mole base T_c , base T_m	15.15	39	2.59	36	2.38
8	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	80.45	51	0.63	60	0.75
9	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	75.04	63	0.84	56	0.75

Table 6: Hydrogen production

measurements. Figure 39 presents the percent of lithium reacted in the alloy calculated from hydrogen measurements by mass spectrometer (sample “B”: five minutes after the impact) and the titration method from the water solutions.

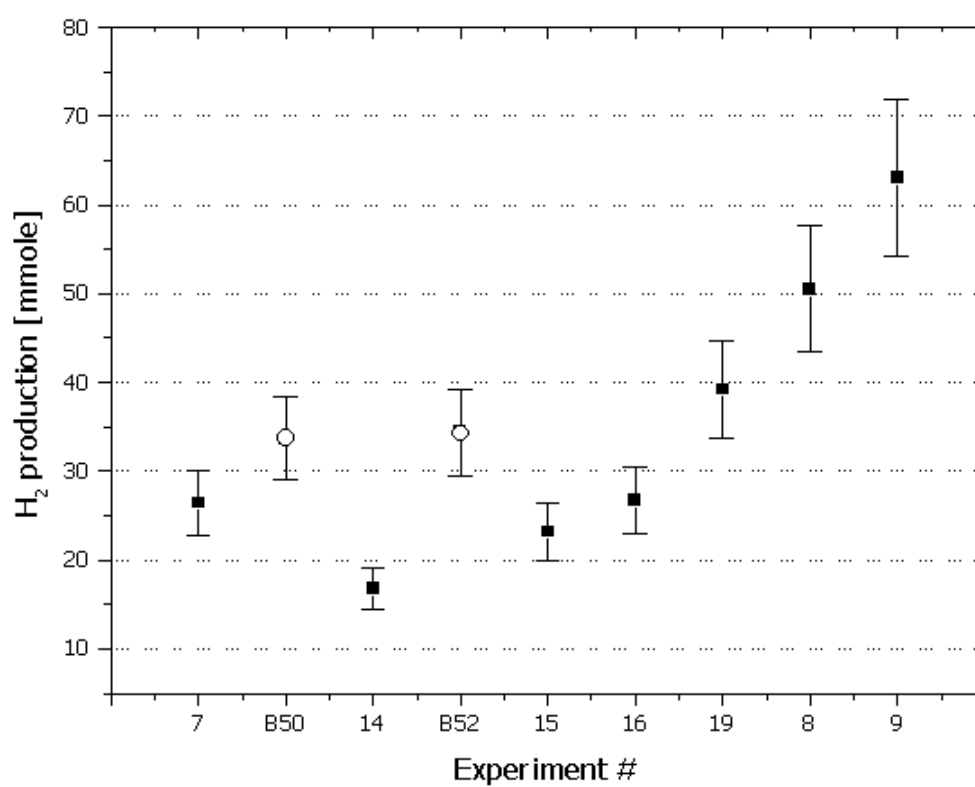


Figure 38: Hydrogen production from mass spectrometer measurements.

Expt#	Sample	Li-mass [g]	Percent Li-reacted [%]		
			Mass Spec "A"	Mass Spec "B"	Titration
7	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , base T_m	0.82	40.9	44.2	63.8
B-50	Pb ₈₃ Li ₁₇ base T_c , base T_m	0.82	-	55.9	73.0
14	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , base T_m	0.82	23.7	28.5	62.7
B-52	Pb ₈₃ Li ₁₇ high T_c , base T_m	0.86	-	55.8	73.6
15	Sn ₇₅ Li ₂₅ fix Li-mole base T_c , high T_m	0.79	32.6	40.6	71.0
16	Sn ₇₅ Li ₂₅ fix Li-mole high T_c , high T_m	0.76	46.0	48.5	49.5
19	Sn ₅₀ Li ₅₀ fix Li-mole base T_c , base T_m	0.84	63.2	65.1	59.8
8	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	1.70	21.3	41.3	54.4
9	Sn ₇₅ Li ₂₅ fix volume base T_c , base T_m	1.43	51.4	61.1	54.5

Table 7: Percent of Li-reacted

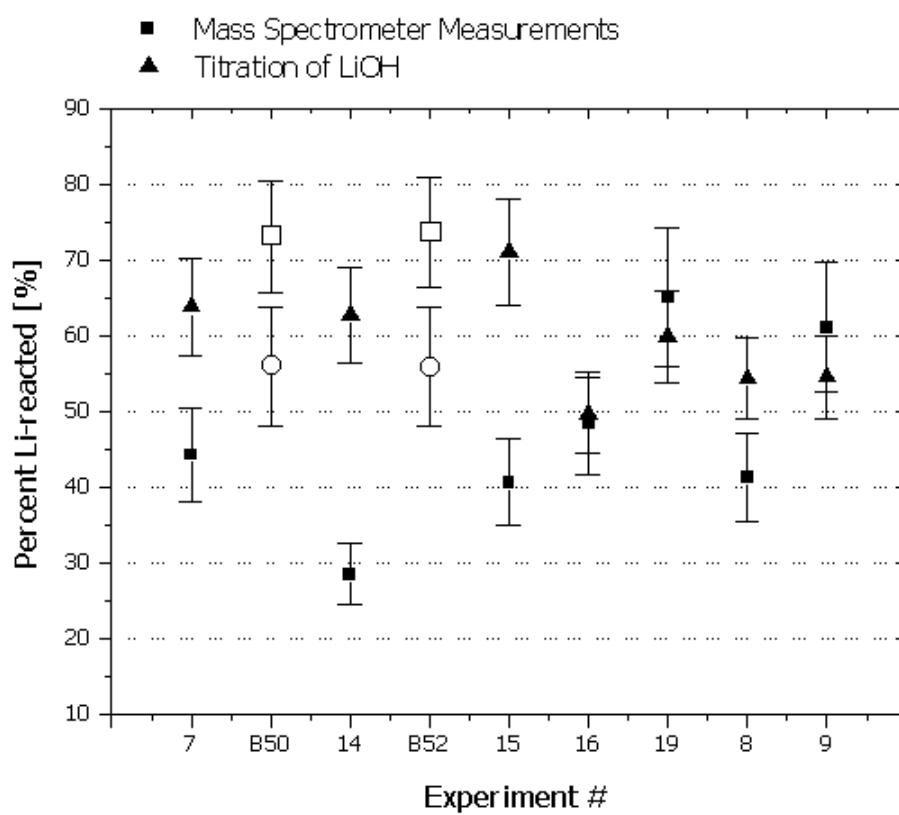


Figure 39: Percents of lithium reacted calculated from the gas samples and the water samples.

Chapter 5

Conclusions

Preliminary experiments with an empty crucible and pure tin have been conducted, to ensure that the dynamic pressure measurements are consistent with those of Vukovic [11] and Nelson et al. [7]. The dynamic pressure measurements indicate good agreement with past data and can be reviewed in Figures 19, 20, 21, 22 and 23. The results of the alloy experiments indicate that the alloy $\text{Sn}_{75}\text{Li}_{25}$ is qualitatively similar in behavior to $\text{Pb}_{83}\text{Li}_{17}$ in terms of hydrodynamic and chemical interactions with water. The dynamic pressures measured are also quite similar to that of the lead alloy (Figure 24, 25). A dynamic pressure trace from the $\text{Sn}_{50}\text{Li}_{50}$ experiment (Figure 32) is also similar to that of the $\text{Sn}_{75}\text{Li}_{25}$ experiment (Figure 26), except for a slightly higher impact pressure associated with $\text{Sn}_{50}\text{Li}_{50}$ due to greater hydrogen production (Figure 38).

The impulse ratio of the experimental to the theoretical value of the first interaction pulse is a good indicator of the energetics of the reactions. Figure 36 indicates that the impulse ratio of the non-reactive experiments are all less than 1.0 due to losses. In contrast, all the reactive experiments produce an impulse ratio greater than 1.0 due to thermic interaction. From the same plot, one can see that for higher coolant and/or molten metal temperatures, the higher impulse ratio produced, implying a stronger interaction. The work ratio of the compression work to expansion work of the gas expansion vessel is another representation of the energetics of the reaction. As one might expect, the work ratio has a trace similar to that of the impulse ratio as shown in Figure 37. The plot does not distinguish between differences in each experiment as

clearly as that of the impulse ratio since the volume of the gas expansion vessel cannot be measured precisely, which leads to higher uncertainty of the work calculation. However, the plot of the work ratio indicates the overall trace of the energy generated in the experiments. The lowest work ratio belongs to two of the empty crucible experiments and becomes higher in the experiments containing the reactive alloy.

Measurement of hydrogen production provides confirmation that we have a more energetic reaction in the reactive experiments of the Sn-Li alloy. Figure 38 indicates clearly that more hydrogen has been produced when the coolant and/or the molten temperatures are higher. As expected by Nelson in the Pb-Li experiments, most of the hydrogen was generated during the first 5 minutes after the impact [7]. The second measurements of hydrogen obtained from the titration method from the water solution after impact agree with the mass spectrometer measurements. The percent of Li-reacted has been calculated from the hydrogen measurement based on the most probable chemical reaction between lithium and water. It was found that the reaction rate of the Li alloys are remarkably less than that of the pure lithium.

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Appendix A

Procedure for Production of Alloy

Preparation before the synthesizing

1. Remove all the dust in the glove box with vacuum cleaner.
2. Attach the band electric heater to the reactor and put inside the fire bricks set.
3. Insert a thermocouple for the heater between the reactor and the band heater.
4. Set the reactor on the stand plate and attach the edges of the fire brick with steel angle bars and insert fire a thermocouple for the fire brick between the steel angle bar and the fire brick.
5. Mount the fire brick to the stand plate with steel belts.
6. Firmly attach the reactor set on the rotated stand in the glove box and make sure that the reactor set will not slide down when the stand is rotated.
7. Connect the heater wires to power wires from heater controller, cover the connection with plastic caps.
8. Make sure that the heater works properly by turning on the heater controller to 70% of 120 volts, the ammeter should read around 2.70 to 2.90 amps.
9. Attach the stir rod to the stir motor and make sure that the stir motor works well.
10. Prepare all the necessary materials and equipment in the glove box:

- Tin (Sn) shot in a container
 - Lithium (Li) foil closed in argon (Ar) atmosphere in a well sealed container
 - Reactor cap to pour the molten alloy
 - Graphite molds to cast the alloys
 - Graphite plate to prevent contact between the molten and glove box floor
 - Mold holder
 - Alloy labels with transparent tape to label the cast alloys
 - 1 container with sealed lid for the cast alloys
 - 2 small sample containers with sealed stopper for composition analysis
 - Scissors to cut the lithium foil
 - Scale to measure mass of the materials
 - A big pliers to trim out the overflow cast alloy
 - The crucible that will be used in the water impact experiment
 - Another stainless steel stir rod to mix the molten alloy by hand
 - Convex mirror attached to the stir motor stand to see inside the reactor
 - Thermocouple holder attached to the stir motor stand
 - Thermocouple wire in a small stainless tube to be used in the molten.
11. Measure the necessary amount of Sn shot and put all into the reactor.
 12. Insert the molten thermocouple in the Sn shot in the reactor.
 13. Connect all the thermocouples and make sure that all work properly by testing reading temperatures from the data acquisition unit, all the thermocouple should read room temperature.

14. Open lids of all empty containers to prevent air locked in containers.
15. Close and seal the glove box.
16. Start purging the glove box with Ar gas from a pressurized cylinder by flowing Ar 5 times of the glove box volume while all the outlet valves are opened and the purging sensor is on (takes about an hour and a half at 85 CFH).

Melt and mix the alloy

17. Make sure that the purging sensor of the glove box is still on and it will be on for the entire synthesizing process.
18. Turn on the heater to 70% of 120 volts.
19. Start reading temperatures with the data acquisition unit.
20. When Sn start melting at around 240°C, volume of the Sn in the reactor will decrease until all melted.
21. When all Sn is molten or the molten temperature reaches 350°C, turn on the stir motor.
22. Continue heating the molten and keep stirring the molten Sn.
23. While Sn is being heated, prepare Li strip to put into the reactor.
24. To prepare the Li strips, open the Li container (in the glove box), cut the Li foil into strips (approximately 1.5 cm wide; mass of a Li strip will be around 0.8 to 0.9 g) with scissors and measure mass of Li strips until they reach the necessary amount.
25. When the molten temperature reaches 500°C (melting point of $\text{Sn}_{75}\text{Li}_{25}$ is around 327°C), control the molten temperature constant at 500°C.

26. While the stir rod is turning, slowly put Li strip into the reactor to react with the molten Sn.
27. An exothermic reaction will occur with noticeable red growing light and a little bit of smoke. The molten temperature will rise, may be higher than the heater temperature, and back down to normal within 2-3 seconds.
28. After the reaction is complete, the surface of the alloy in the reactor may become solid, extra stirring by hand with another stir rod may be needed to make all the alloy melt homogeneously and ready for the next Li strip.
29. Add the next Li strip into the reactor. The reaction will occur again and the previous process may be needed.
30. Continue adding the Li strip slowly by repeating process 26 to 29.
31. After all the Li strips are added and completely mixed with the molten alloy in the reactor, turn off the stir motor and remove the stir rod and the molten thermocouple while the heater is still on.
32. Stirring by hand may be needed to make sure that the alloy is mixed homogeneously.

Casting the alloy and taking samples

33. Carefully screw the reactor cap on the reactor about 2 turns and have the pouring tube on the right, the side that the reactor will be rotated.
34. With the mold holder, hold the graphite mold and slowly crank the stand to rotate the reactor toward the mold.
35. Slowly pour the molten alloy to the mold to take to first sample (the top part of the molten alloy).

36. When the molten reaches about a half of the mole, immediately crank the reactor up to stop the flow.
37. Let the alloy sample freeze in the mold for a second and knock the alloy billet out on the graphite plate.
38. Start casting the first alloy billet by pouring the molten to the mold until full, repeat the pouring process until getting the needed number of alloy billets.
39. After the last alloy billet has been made, pour the leftover molten alloy into the mold to take the second sample (the bottom part of the molten).
40. Rotate the reactor back to the right up position and turn off the heater.
41. Leave all the cast alloy cool down on the graphite plate in the glove box.

Finalize and identify the alloy billet

42. With the pliers, trim out the excess mass of the alloy billets to make them fit into the crucible.
43. Measure mass of each alloy billet, place the label and keep the records.
44. Put the identified alloy billets into the alloy container and close the lid when all is done.
45. The purging sensor of the glove box may be turned off now.

Appendix B

Analyses of Alloy Production

Alloy Mass

Before calculating the necessary mass of each element to be mixed in the reactor, the density of the final alloy is needed. The density was estimated based on mass fraction as

$$\rho_{alloy} = X_{Li}\rho_{Li} + X_{Sn}\rho_{Sn}$$

where ρ is density and X is mass fraction which is defined as

$$X_a = \frac{n_a M_a}{n_a M_a + n_b M_b}$$

where n and M are mole number and molar mass (atomic weight) of the element respectively. The mass fractions of each element in the alloys are shown in Table 9

Properties of lithium and tin used in the calculation are shown in Table 8.

Properties	Lithium (Li)	Tin (Sn)
Atomic Number	3	50
Atomic Weight	6.91 g/mole	118.71 g/mole
Melting Point	180.54°C	231.97°C
Boiling Point	1,342°C	2,270°C
Density at 27°C	0.535 g/cc	7.31 g/cc

Table 8: Properties of the elements

Alloy	Li	Sn
Sn ₇₅ Li ₂₅	1.9117	98.0883
Sn ₅₀ Li ₅₀	5.5240	94.4760

Table 9: Percent by mass of the elements in alloys

Mold Volume

The shape of the cast alloy should be consistent with the crucible which is a cylindrical shape with diameter about 2.0 to 2.2 cm. The mold cannot be a simple straight cylinder since the cast alloy may not be able to knock out of the mold. A small taper angle is needed as shown in Figure 40.

The volume of the mold will be approximately equal to the volume of the cast alloy which can be calculated by integrating an infinitesimal cylinder along the mold height H :

$$\begin{aligned}
 V &= \int_0^H \pi r^2 dh \\
 &= \pi \int_0^H \left(\frac{D}{2} - L \tan \theta \right)^2 dh \\
 &= \pi \int_0^H \left(\frac{D^2}{4} - DL \tan \theta + L^2 \tan^2 \theta \right) dh \\
 &= \pi H \left(\frac{D^2}{4} - \frac{DH}{2} \tan \theta + \frac{H^2}{2} \tan^2 \theta \right).
 \end{aligned}$$

Since $H = L \cos \theta$, the volume can be calculated as,

$$V = \pi L \cos \theta \left(\frac{D^2}{4} - \frac{DL}{2} \sin \theta + \frac{L^2}{3} \sin \theta \right).$$

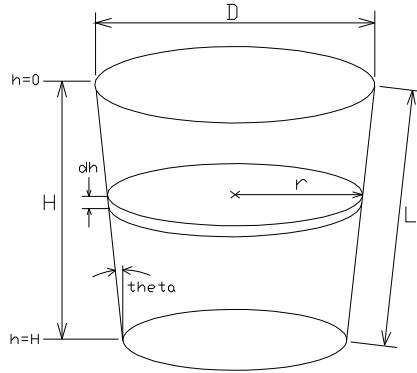


Figure 40: Parameters of the mold

Composition Analysis Results

The core samples of the alloy were sent to the University of Wisconsin Soil Plant Analysis Laboratory¹ for composition analysis. There were two samples taken from each alloy, Sn₇₅Li₂₅ and Sn₅₀Li₅₀, one from the top first inch of the alloy in the reactor and the other from the bottom in order to analyze homogeneousness of the alloy as additional information. Samples s-1 and s-2 were taken from Sn₇₅Li₂₅ from the top and the bottom respectively. Samples s-3 and s-4 were taken from the top and the bottom of Sn₅₀Li₅₀ respectively. Each sample was analyzed twice to minimize errors. The analysis results from the laboratory are shown quantitatively in Table 10.

In summary, the average atom percent of Li and Sn in Sn₇₅Li₂₅ is 27.49 and 72.39 with standard deviation of 2.7 and 2.3 respectively. The average atom percent of Li and Sn in Sn₅₀Li₅₀ is 49.61 and 50.09 with standard deviation of 2.3 and 2.1 respectively.

Note: Atom percent can be calculated from weight percent using the formula below,

$$atom\%_{Li} = \frac{\frac{wt\%_{Li}}{MM_{Li}}}{\left(\frac{wt\%_{Li}}{MM_{Li}} + \frac{wt\%_{Sn}}{MM_{Sn}} + \frac{wt\%_{Cr}}{MM_{Cr}} + \frac{wt\%_{Ni}}{MM_{Ni}}\right)} \times 100$$

where MM_{Li} is molecular mass of Li.

¹Soil Plant Analysis Laboratory, 5711 Mineral Point Rd. Madison, WI 53705-4453
Phone:(608)262-4364 Fax:(608)263-3327 <http://riprock.soils.wisc.edu>

Sample	Li		Sn		Cr		Ni		Fe	total
No.	wt%	atom%	wt%	atom%	wt%	atom%	wt%	atom%	wt%	wt%
s-1	2.20	30.31	85.95	69.38	0.04	0.07	0.15	0.24	< 0.2	88.33
s-1	2.06	27.23	94.14	72.60	0.02	0.04	0.08	0.13	< 0.2	96.31
s-2	1.77	24.92	90.92	75.04	0.02	0.04	0.00	0.00	< 0.2	92.70
s-2	2.01	27.37	91.04	72.53	0.02	0.04	0.03	0.05	< 0.2	93.11
s-3	5.88	52.16	91.91	47.70	0.02	0.02	0.11	0.12	< 0.2	97.92
s-3	5.67	51.04	92.86	48.85	0.02	0.03	0.08	0.09	< 0.2	98.63
s-4	5.12	47.69	94.99	51.78	0.02	0.03	0.45	0.50	< 0.2	100.59
s-4	5.00	47.53	93.62	52.02	0.03	0.04	0.36	0.41	< 0.2	99.02

Table 10: Composition analysis results from Soil Plant Analysis Laboratory:
Analyzed on January 31st, 2001.

Appendix C

Operational Procedure for Shock Tube Experiment

Preparing before the test

1. Clean and dry the lower section of the tube, including all the connected pipe, thoroughly using pressurized air and heat gun.
2. Wrap around the sharp edge of the top of the lower section with Teflon pipe thread tape to lubricate the pipe nut that will be screwed into the middle section.
3. Apply vacuum grease to the top of the lower section and the bottom of the middle section where the diaphragm will be installed.
4. Place a Kapton disc on the bottom of the middle section. The disc should stick to the grease under the middle section.
5. Connect the top of the lower section to the bottom of the middle section where the diaphragm has been placed. Carefully screw in the lower section to the middle section until tight.

Caution: The diaphragm can be broken if the lower section is screwed too tight.

6. Fill the water bath unit with distilled water to ensure that there will be enough water to run the test.
7. Check that the outlet port of the water bath unit is connected to the “inlet”

port of the shock tube near valve#4 and inlet port of the water bath unit is connected to the “outlet” of the tube near valve#1.

8. Start running water to the middle section by turning on the water bath unit, closing valve#2 and 5 and opening valve#4, 3 and 1.
9. Water will run through the middle section above the diaphragm. Make sure that the diaphragm is not broken or leaks by looking underneath the lower section using a flashlight to see whether there is water leaking from the diaphragm.
10. Place a clean paper towel under the lower section and let the water run through the tube for 5-10 minutes to ensure that there is no water dripping onto the paper towel.

Note: If there is even a drop of water dripping from the diaphragm, reinstallation of the diaphragm will be needed, which means the entire processes starting from #1 will be repeated.

11. Get the test material ready in a crucible in the glove box and seal with a rubber plug to prevent alloy contact with air. Record weight of the alloy with crucible, the rubber stopper and the copper gasket before the test.
12. Connect the argon line from an argon cylinder to the port near valve#6. Make sure that there is enough argon in the cylinder (at least 400 psi).
13. At the lower section, close the vacuum gauge port between valve#6 and 7 with a smaller rubber plug. Open valve#6 and 7 and let argon flow through the lower section and out where the crucible will be installed.
14. Insert the crucible into the crucible housing. Under the lower section where argon is flowing out, open the rubber stopper of the crucible and put a copper

gasket on the crucible then slowly screw in the crucible housing to the lower section while continuing argon flow.

15. When there is no argon leaking out through the thread while the crucible is being screwed, slowly open valve#8 to relieve a small amount of argon from the lower section to prevent it from overpressurized.
16. When the end of the thread is reached, extend tightening the crucible with an extended wrench to squeeze the copper gasket in order to make a good seal for the reaction chamber.

Caution: Squeeze the copper gasket as much as possible but do let the lower section move, otherwise the diaphragm will be twisted and broken. While squeezing the copper gasket, the crucible should not be able to turn easily more than half a turn. If so, it is possible that the copper gasket is out of center and a good seal cannot be established.

17. Close valve#6, 7 and 8 and stop flowing argon from the supply then unplug the rubber stopper between valve#6 and 8. Before screwing in the vacuum gauge, wrap the thread of the gauge with Teflon tape and apply a small amount of vacuum grease over the tape.
18. Connect the vacuum port near valve#8 to a mechanical vacuum pump then turn on the pump.
19. While the vacuum pump is running, fully open valve#8 and 7 (valve#6 is still closed). Read the pressure data from the vacuum gauge to see if the reaction chamber can be evacuated down to about 0.3 torr. This process may take several minutes to get the pressure down.
20. Test how well the reaction chamber can hold the vacuum by closing valve#8, stop the vacuum and see if the pressure inside the chamber is not increasing very

fast. It is very difficult to make a perfect seal to the chamber. Once the valve is closed the pressure should be slowly raised at the rate of about 0.01 torr per second from 0.3 torr to about 0.7 torr. At this rate, the chamber will be able to hold pressure at about 1 torr for about 2-3 minutes which is long enough for the experiment.

21. Once the vacuum seal is satisfied, slowly flow argon to the chamber to about 20 torr then close valve#7 and 8 to lock the gas in the chamber during the heating process.

Running the test

22. Set up the radiant electric heater around the crucible. Hook up the thermocouple wire of TC5 and make sure that it is reading correctly. Start heating the crucible using 70% of 120V of both heaters.
23. The water is still running through the middle section while the desired water temperature has been set.
24. Double check all the settings of the charge amplifier e.g. gains, ranges (for PCB charge amp, set the period switch to “short” and main switch to “OPR”).
25. Prepare two bottles of the gas sample by evacuating these two bottles on the piping portion of the mass spectrometer for 2-3 minutes then install the bottles to the multi-way valve at the top of the expansion vessel.
26. Once the desired temperature of the molten metal has been reached, reduce the heating power to maintain the constant temperature of the molten.
27. Open valve#7 and 8 and start evacuating the reaction chamber again to the lowest possible pressure.

28. Close valve#4 then valve#1 and open valve#2. Adjust water level in the tube by slowly opening valve#4 until the water level reaches the red line of the water scale tube by the expansion vessel, then close valve#4, 3 and 2.
29. Switch the multi-way valve to “open” position and open the solenoid valve and let argon pass through the expansion vessel for 30 second in order to purge air out of the expansion vessel. Switch the multi-way valve to “close” and stop flowing argon.
30. Slowly flow argon to the reaction chamber to about 0.9-1.0 torr then immediately close valve#7 and 8 to lock and isolate the chamber. Turn off the vacuum pump.
31. Set oscilloscope and computer to be ready to record data from the transducers after a trigger from PT0.
32. Make sure that the valves#1, 2, 3, 7 and 8 are closed tightly.
33. Start pressurizing the expansion vessel by opening the pressure regulator on the argon cylinder and keep watching pressure in the vessel from either pressure regulator or computer screen.
34. When the pressure reads about 100 psi, slowly increasing pressure until the diaphragm ruptures with a noticeable sound “bang”.
35. The solenoid valve will be closed automatically when the transducer is PT0 triggered. All the pressure data should be recorded within 0.1 second after the impact.
36. The pressure regulator can be now closed. Turn off the radiant electric heater.

Collect samples after the test

37. Take the first gas sample by switching the multi-way valve to position “A” for the first gas sample bottle then open a valve of the bottle to take the sample then close the valve.
38. Wait for about 5 minutes before taking the second gas sample. To take the second sample, switch the multi-way valve to position “B” then open the valve of bottle “B” to take the sample then close.
39. Relieve the rest of the gas in the expansion vessel by switching the multi-way valve to “open”. Wait until the gas flowing sound diminishes then disassemble the gas sample bottles.
40. At the mass spectrometer, record the background data in the vacuum chamber at the leak tight position of the variable leak valve before reading the sample.
41. To analyze the gas sample, connect the bottle to the variable leak valve and evacuate the piping portion for 2-3 minutes. Isolate the pipe to the atmosphere, turn off the vacuum pump and open the valve of the sample bottle.
42. Slowly open the variable leak valve to let the gas sample flow through the vacuum chamber and maintain the total pressure constant at 5.0×10^{-5} mbar. Then start recording the partial pressure data of the sample.
43. Repeat the same procedure beginning from reading the background data for the second sample.
44. The liquid shock tube will be left to cool down for 4-8 hours before taking the lower section and crucible apart.
45. All the water from the reaction will be collected by opening valve#3 and 5 and letting the water drain to a clean bucket.

46. Prepare the water bucket under the reaction chamber then use the extended wrench to unscrew the crucible. Some amount of water from the lower section will drain to the bucket. Pour the residual water in the crucible into the bucket.
47. Stir the water sample in the bucket well and measure the total volume of the water, then save only 600 cc of the aqueous solution sample in a glass jar with screw sealed lid.
48. To collect the alloy debris, knock out the debris in the crucible onto a layer of clean paper towels. The debris can remain in the lower part of the middle section of the tube from the explosion. This debris will also be dried on the paper towels.
49. Let the debris dry on the paper towels and dry the crucible and the copper gasket with heat gun.
50. Measure weight of the debris, dried crucible, copper gasket and the rubber stopper to compare with the weight before the test.
51. Save the debris in a clear, non-reactive plastic container with lid. The tested crucible and copper gasket will also be saved.
52. Disconnect the argon line from the supply to the lower section and the vacuum line, remove the vacuum gauge, then unscrew the lower section from the middle section.
53. Move the lower section to a stand to clean and dry and be ready for the next test.

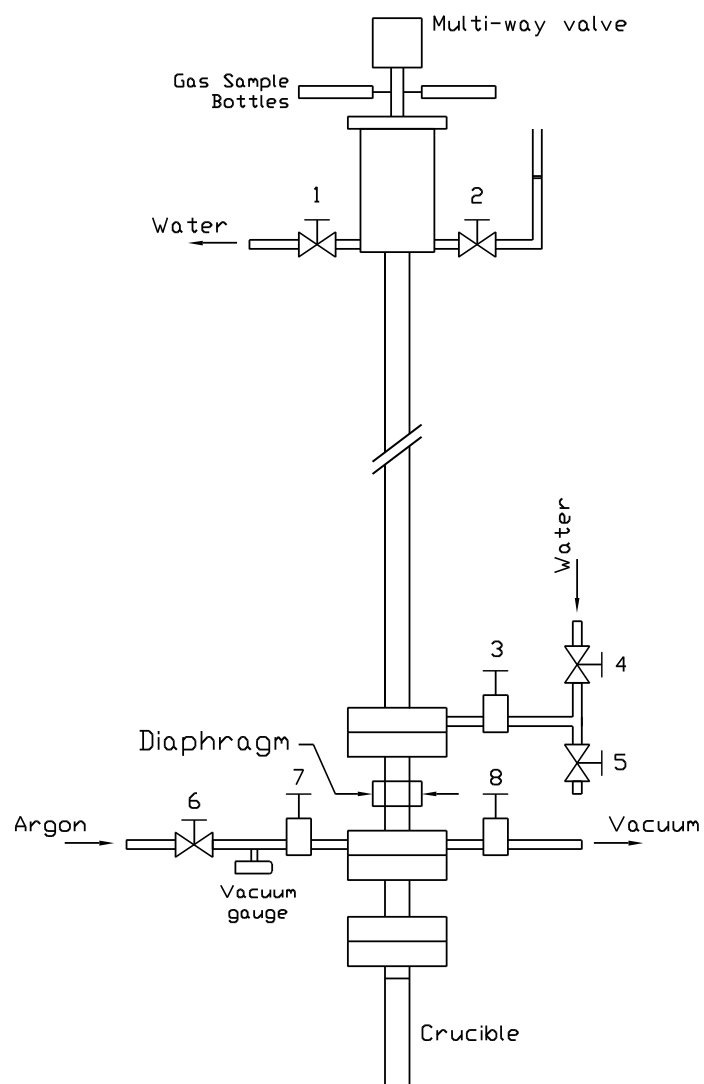


Figure 41: Schematic of the shock tube illustrates the piping system and location of valves.

Appendix D

Estimation of Measurement Uncertainties

Total uncertainty of a particular parameter, R , can be calculated as a function of uncertainty of each measurement, m_i , by the following equation

$$\delta R = \sum_{i=1}^n \left| \frac{\partial R}{\partial m_i} \right| \overline{\delta m_i} \quad (4)$$

where n is a total number of measurements relevant to calculate the parameter R and $\overline{\delta m_i}$ is average uncertainty of measurement m_i .

Uncertainty of Impulse Ratio

The impulse ratio can be calculated using the equation

$$R = \frac{I^{expt}}{I^{th}} = \frac{I^{expt}}{2(p_{inj} + \rho_c H_c g) t_{imp}^{th}}$$

where $t_{imp}^{th} = \sqrt{\frac{2H_{fall}\rho_c H_c}{p_{inj} + \rho_c H_c g}}$ is as in equation (2). The following equations will be combined in equation (4) to calculate the uncertainty of the impulse ratio.

$$\begin{aligned} \frac{\partial R}{\partial p_{inj}} &= -\frac{I^{expt}}{4(p_{inj} + \rho_c H_c g)^2 t_{imp}^{th}} \\ \frac{\partial R}{\partial H_c} &= -\frac{(p_{inj} + 2\rho_c H_c g) I^{expt}}{4(p_{inj} + \rho_c H_c g)^2 H_c t_{imp}^{th}} \\ \frac{\partial R}{\partial H_{fall}} &= -\frac{I^{expt}}{4(p_{inj} + \rho_c H_c g) H_{fall} t_{imp}^{th}} \end{aligned}$$

Expt#	N	δR
2	2127	0.0836
B-25	200	0.0496
3	2219	0.0887
B-31	174	0.0468
4	4058	0.1515
B-35	624	0.1315
7	3815	0.1378
B-50	344	0.0885
14	3801	0.1502
B-52	480	0.1208
15	3878	0.1517
16	3937	0.1567
19	3815	0.1491
8	3115	0.1805
9	3996	0.1662

Table 11: Uncertainty of impulse ratio

$$\frac{\partial R}{\partial I^{expt}} = -\frac{1}{2(p_{inj} + \rho_c H_c g) t_{imp}^{th}}.$$

The following average uncertainty of measurements were used in this report; $\overline{\delta p_{inj}} = 5$ psi, $\overline{\delta H_c} = 0.01$ m, $\overline{\delta H_{fall}} = 0.005$ m. $\overline{\delta I^{expt}}$ has to be treated as special since it is not a direct measurement but it is calculated from $I^{expt} = \sum (p \cdot \Delta t)$; then

$$\begin{aligned} \overline{\delta I^{expt}} &= \sum_N (\overline{\delta p} \cdot \Delta t) \\ &= (\overline{\delta p} \cdot \Delta t) \cdot N \end{aligned}$$

where N is the number of dynamic pressure data that is used to calculate I^{expt} and Δt is the time between each pressure measurement which is 0.00002 s for our experiments and 0.000125 s for Nelson's experiments [7]. $\overline{\delta p}$ is 2% of full scale which is approximately 20 psi. Table 11 presents the number of pressure data used and total uncertainty of the impulse ratios.

Uncertainty of Work Ratio

From equation (3), the work ratio can be determined from

$$R = \frac{W^{comp}}{W^{expd}} = \frac{p_3 V_3 - p_2 V_2 + mgH_{fall}(1 - \gamma)}{p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma)}.$$

The following equations will be combined in equation (4) to calculate the uncertainty of the work ratio:

$$\begin{aligned} \frac{\partial R}{\partial p_1} &= \frac{V_1 (p_3 V_3 - p_2 V_2 + mgH_{fall}(1 - \gamma))}{(p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma))^2} \\ \frac{\partial R}{\partial V_1} &= \frac{p_1 (p_3 V_3 - p_2 V_2 + mgH_{fall}(1 - \gamma))}{(p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma))^2} \\ \frac{\partial R}{\partial p_2} &= \frac{V_2 (p_3 V_3 - p_1 V_1 + 2mgH_{fall}(1 - \gamma))}{(p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma))^2} \\ \frac{\partial R}{\partial V_2} &= \frac{p_2 (p_3 V_3 - p_1 V_1 + 2mgH_{fall}(1 - \gamma))}{(p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma))^2} \\ \frac{\partial R}{\partial p_3} &= \frac{V_3}{p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma)} \\ \frac{\partial R}{\partial V_3} &= \frac{p_3}{p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma)} \\ \frac{\partial R}{\partial H_{fall}} &= \frac{mg(\gamma - 1) (p_1 V_1 - 2p_2 V_2 + p_3 V_3)}{(p_2 V_2 - p_1 V_1 + mgH_{fall}(1 - \gamma))^2}. \end{aligned}$$

The following average uncertainty of measurements were used in this report:

$$\begin{aligned} \overline{\delta p_1} &= 0.5 \text{ psi} & \overline{\delta V_1} &= 2.5 \times 10^{-5} \text{ m}^3 \\ \overline{\delta p_2} &= 0.5 \text{ psi} & \overline{\delta V_2} &= 2.5 \times 10^{-5} \text{ m}^3 \\ \overline{\delta p_3} &= 0.5 \text{ psi} & \overline{\delta V_3} &= 2.5 \times 10^{-5} \text{ m}^3. \end{aligned}$$

Appendix E

Instrumental Records

All instrumental records including experimental configuration sheets of every experiments are presented in the following pages. [Not included in electronic version.]