

# SANDIA REPORT

SAND2001-0616

Unlimited Release

Printed by [unclear] at [unclear]

## Safety Analysis of High Pressure $^3\text{He}$ -filled Micro-channels for Thermal Neutron Detection

M.S. Derzon, R.F. Renzi, S.M. Ferko, and P. C. Galambos

Prepared by  
Sandia National Laboratories  
Albuquerque, New Mexico 87185 and Livermore, California 94550

Sandia is a multiprogram laboratory operated by Sandia Corporation,  
a Lockheed Martin Company, for the United States Department of Energy's  
National Nuclear Security Administration under Contract DE-AC04-94AL85000.



Issued by Sandia National Laboratories, operated for the United States Department of Energy by Sandia Corporation.

**NOTICE:** This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government, nor any agency thereof, nor any of their employees, nor any of their contractors, subcontractors, or their employees, make any warranty, express or implied, or assume any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represent that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government, any agency thereof, or any of their contractors or subcontractors. The views and opinions expressed herein do not necessarily state or reflect those of the United States Government, any agency thereof, or any of their contractors.



"SAND200: /9649

Wprko kgf "Tgrgcug"

Printed P qxgo dgt "422:

# Safety Analysis of High Pressure <sup>3</sup>He-filled Micro-channels for Thermal Neutron Detection

M.S. Derzon, R.F. Renzi, S.M. Ferko, P.C. Galambos

National Laboratories

P.O. Box 5800

Albuquerque, New Mexico 87185-MS1196

## Abstract

This document is a safety analysis of a novel neutron detection technology developed by Sandia National Laboratories. This technology is comprised of devices with tiny channels containing high pressure <sup>3</sup>He. These devices are further integrated into large scale neutron sensors. Modeling and preliminary device testing indicates that the time required to detect the presence of special nuclear materials may be reduced under optimal conditions by several orders of magnitude using this approach. Also, these devices make efficient use of our <sup>3</sup>He supply by making individual devices more efficient and/or extending the our limited <sup>3</sup>He supply. The safety of these high pressure devices has been a primary concern. We address these safety concerns for a flat panel configuration intended for thermal neutron detection.

Ballistic impact tests using 3 g projectiles were performed on devices made from FR4, Silicon, and Parmax materials. In addition to impact testing, operational limits were determined by pressurizing the devices either to failure or until they unacceptably leaked. We found that 1) sympathetic or parasitic failure does not occur in pressurized FR4 devices 2) the Si devices exhibited benign brittle failure (sympathetic failure under pressure was not tested) and 3) the Parmax devices failed unacceptably. FR4 devices were filled to pressures up to  $4000 \pm 100$  psig, and the impacts were captured using a high speed camera. The brittle Si devices shattered, but were completely contained when wrapped in thin tape, while the ductile FR4 devices deformed only. Even at 4000 psi the energy density of the compressed gas appears to be insignificant compared to the impact caused by the incoming projectile. In conclusion, the current FR4 device design pressurized up to 4000 psi does not show evidence of sympathetic failure, and these devices are intrinsically safe.

## **ACKNOWLEDGMENTS**

Sandia National Laboratories is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy under contract DE-AC04-94AL85000.

We wish to acknowledge those who have contributed to this project. First we wish to acknowledge the financial support of DTRA for this safety study. In addition, this list includes but is not limited to Kevin Seager, Tom Weber, Dusty Rhoades, Mark Grohman, Jeff Lantz, Catalina Rivas, Mark Claudnic, Dave Zanini, Dan Yee, Roger Shrouf, Robert Anderson, Dave Myers, Carol Sumpter, Sita Mani, Keith Ortiz, Tom Zipperian, of Sandia. The Sandia Seniors council sponsored the first investment in the technology through a Seniors Council LDRD. We gratefully acknowledge Bart Ebbinghaus, of NA-12/NA-47, and Clint Hall for continuing the development effort regarding the basic physics of this class of devices.

## CONTENTS

1	Executive Summary .....	8
2	Introduction.....	10
	2.1 Principles of Operation .....	10
3	Methodology .....	15
	3.1 Safety Comparison of Proposed Devices to Existing Technology .....	16
	3.2 Finite Element Analysis of Capillaries .....	18
	3.2.1 FR4 Devices .....	18
	3.2.2 Silicon Devices.....	19
	3.2.3 Parmax devices.....	20
	3.3 Projectile and Burst Tests .....	22
	3.4 Experimental .....	23
	3.4.1 Facilities .....	23
	3.4.2 Set-up Description & Detail .....	24
	3.4.3 Target Devices and Arrayed Assemblies .....	26
	3.4.4 Data acquisition equipment .....	26
4	DOT Requirements .....	27
5	Results, Discussion and Conclusions.....	28
	5.1 General Overview & Common Results .....	28
	5.2 FR4.....	28
	5.3 Silicon .....	30
	5.4 Parmax .....	33
	5.5 General Conclusions .....	35
	6.0 Future Developments .....	35
7	Appendix.....	36
8	Distribution .....	43

## FIGURES

Figure 1. Simple model estimate of efficiency as a function of capillary area fraction $f_c$ . Individual curves show effect versus $^3\text{He}$ pressure. The predicted design space has a maximum area fraction of about 80% at 1000 psi, falling to 50-70% at 5000 psi in FR4 or about 50% at 10,000 psi in a 0.7 mm thick Si wafer. Use of $f_b=0.2$ allows for conservative estimates from this model.....	12
Figure 2. Dependence of expected intrinsic device efficiency using a fixed area fraction of $f_c=0.5$ at various pressures. Neutron albedo, $f_b$ , is typically about 15% for fast neutrons and 80% for thermal neutrons. Depending on configuration and source spectrum these numbers can vary. Mathematically the model goes above $f_b=0.8$ however the model assumptions are expected to be inaccurate above that point. The point at $f_b=1$ is shown merely for completeness.....	13
Figure 3. Schematic drawing of high efficiency capillaries arranged in a simple array.....	14
Figure 4. a) Backside photo of 9-plex FR4 array showing gas fill capillaries and.....	15
Figure 5. 3D image of stress versus location in ANSYS model of spiral capillary embedded in Si wafer. 10,000 psi of gas fills the capillary. ....	20
Figure 6. 2D model of Si channel. This cross section image provides a high resolution look at the stress in the corners.....	20
Figure 7. Stress distribution around a spiral channel machined .....	21
Figure 8. Stress distribution on overlay of Parmax device. ....	21
Figure 9. Projectile Impact Test Set-up. ....	23
Figure 10. a)High pressure test lab, SNL Livermore, b)Overview of projectile impact test set-up. ....	24
Figure 11. CAD rendering of projectile generator.....	25
Figure 12. Projectile with retaining pin cross hole 1. Projectile weighs 3.5 g.....	25
Figure 13. a)Projectile gun system & catcher, b) Close up of projectile generator.....	26
Figure 14. Successive images of projectile impacting array of 9 FR4 unpressurized devices. Simple double sided tape attached the devices to the support.....	29
Figure 15. Successive images of projectile impacting array of nine FR4 pressurized devices. Devices were bolted to the support plate. ....	29
Figure 16. Successive images of projectile impacting arra of nine 2x2 cm silicon devices.....	30
Figure 17. Successive images of projectile impacting array of 9 2x2 silicon devices.....	31
Figure 18. a) Successive images of projectile impacting silicon device with yellow duct tape, b) Successive images of projectile impacting silicon device with carbon fiber weave, c) Successive images of projectile impacting silicon device with clear tape reinforcement.....	32
Figure 19. Successive images of projectile impacting array of nine pressurized Parmax devices. ....	34

## TABLES

<b>Table 1. Comparison of relative safety concern for various common threats.</b>	<b>17</b>
---	-----------

## NOMENCLATURE

$^3\text{He}$	An isotope of Helium consisting of 2 protons and 1 neutron in the nucleus
atm	atmospheres
dB	decibel
CFR	Code of Federal Regulations
DOD	Department of Defense
DOE	Department of Energy
DOT	Department of Transportation
FEA	Finite element analysis
FR4	Flame Retardant 4, a type of material used for printed circuit boards
Parmax	Self-reinforced polymer (also called Tecamax)
ICF	Inertial Confinement Fusion
LTCC	Low-temperature Co-Fired Ceramic
MeV	Million electron volts
psi	Pounds per square inch
Si	Silicon, Also refers to silicon wafers.
SNL	Sandia National Laboratories
SNM	Special Nuclear Material
STP	Standard Temperature and Pressure
TOF	Time of flight
WMD	Weapons of mass destruction

## 1 EXECUTIVE SUMMARY

Sandia National Laboratories is developing a novel neutron detection technology using ultrahigh pressure  $^3\text{He}$  in tiny channels contained within individual subcomponent devices, to be further arrayed and integrated into large scale neutron sensors.<sup>1,2</sup> Modeling and preliminary device tests indicate that the time required to detect the presence of special nuclear materials may be reduced by several orders of magnitude with this approach, and that safety concerns with high pressure gas can be alleviated.

Historically, concerns for large scale neutron detection systems utilizing high-pressure Helium-3 pertain to safety during transport. Preliminary analysis and testing of micro-channel devices indicates that due to the small size of each micro-channel, and the inherent strength of the materials used for these devices, a detection system made with such devices would be safe for transport and field use.

The work reported here was performed to experimentally validate these preliminary results, to explore the safety aspects of the individual subcomponent devices as assembled in expected detection configurations, and perhaps most importantly, to assist in programmatic decisions. We performed basic projectile testing to understand the potential safety issues as described below and develop a methodology for safety certification.

Four key aspects to the work, and abbreviated results were as follows:

- 1) Projectile tests on unfilled devices fabricated from promising substrate technologies (FR4, machined Parmax, and lithographically processed Si) were performed to determine substrate-specific failure characteristics.
- 2) Projectile tests and burst tests on pressurized devices were performed to determine channel-specific failure characteristics and maximum allowable working pressures. These results are also substrate-dependent, but could help separate material-of-construction issues from channel geometry and layout issues.
- 3) A computational analysis of the three styles of devices was performed in order to confirm the structural integrity and identify possible failure modes or locations. We used standard cylinder/end plate formulas and finite element analysis (FEA) for modeling.



- 4) Analysis of results and comparison with DOT guidance on transportation guidelines performed to help qualify and quantify the proposed devices relative to existing DOT-approved articles.

The FR4 devices held pressure and upon impact they did not sympathetically fail nor shatter. The Si did not hold pressure and sympathetic failure could not be observed. Unpressurized Si devices held together well after impact and remnants were easily captured. The Parmax devices shattered and have been dropped from consideration.

It is concluded that both the individual subcomponent devices and large scale neutron sensor devices which they comprise can be made safe and DOT compliant. It should be noted that there are important advantages to Si devices and Si should continue to be considered after the bonding problem is solved., Detailed results and discussion of all methods and results are included in Sections 3 & 4.

## 2 INTRODUCTION

Neutron sensor systems are required to provide critical data for SNM detection and characterization of nuclear devices. Desirable and critical measurements and device criteria include:

1. Neutron counting with large dynamic range in count rate
2. High thermal neutron detection efficiency
3. Gamma insensitivity
4. Neutron Coincidence detection for background veto (single hit detector mode)
5. Appropriate ergonomics (compact, low weight, easy to handle, low microphonics)
6. Neutron imaging capability

Present detector systems for these measurements, which include scintillators coupled to photomultiplier tubes,  $^3\text{He}$  gas proportional tubes, scintillating fiber arrays, diamond photoconductive detectors and other systems, have their limitations with regards to sensitivity, time response, energy resolution, spatial resolution and background rejection.

An innovative  $^3\text{He}$  high-pressure gas detection system made possible by utilizing Sandia expertise in micro-electrical mechanical fluidic systems is proposed, and has many beneficial performance characteristics with regards to making these neutron measurements in environments such as ports, borders, moving vehicles, etc.

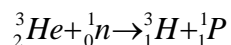
Neutron detection systems utilizing high-pressure Helium-3 gas have traditionally been problematic for field use due to safety concerns during transport. Preliminary analysis and testing of micro-channel devices indicates that the small size of each micro-channel and the inherent strength of the materials used for these devices provides for detection systems that are substantially safer and with higher performance than existing systems, especially during transport and field use. This document describes the safety study and analysis of these channels fabricated in three different materials. The work included a theoretical analysis, projectile tests of pressurized channels, and analysis of DOT requirements.

The work examines the safety aspects of these devices. Basic safety testing as described below was performed, and as development proceeds, inferences regarding the safety of these devices and requirements and methodology for safety certification are also developed.

### 2.1 Principles of Operation

Helium-3 neutron detectors systems are primarily based on the inelastic nuclear reaction,

#### Equation 2.1



This reaction, which is also indicated by the notation  $^3\text{He}(n,p)$  reaction, is an exothermic reaction having a positive energy release or Q-value of 0.764 MeV.<sup>3</sup> The kinetic energy of the tritium and proton charge particle reaction products includes the rather large 0.764 MeV reaction

energy. The detection of these charge particle products is relatively easy and efficient. This feature coupled with the relatively high thermal neutron interaction cross-section makes this system the basis for a number of sensitive neutron detector schemes. This reaction has no energy threshold and the cross-section is very large at low neutron energies having a thermal neutron cross-section of 5330 barns. For fast neutrons produced in ICF relevant fusion reactions, such as 2.45 MeV neutrons produced in Deuterium on Deuterium ions, or 14.5 MeV neutrons produced in Deuterium on Tritium ions, the  ${}^3\text{He}(n,p)$  cross-sections are ~four orders of magnitude lower, 0.75 and 0.13 respectively. Even so, these cross-sections are high enough to make a relatively sensitive and compact detector for Special Nuclear Material (SNM) detection and battlefield applications.

The basic physics and applications space for these detectors is described in a number of references, including Chandler(2) for basic physics, and Derzon(1) an OUO report with a DOD relevant competitive analysis which includes the details of background veto.

Reducing detection times as well as quantity of detected plutonium are the primary considerations motivating this development work. A key aspect of that reduction is the absolute thermal neutron detection efficiency, especially for detection of shielded Plutonium. Thermal neutron detection efficiency is expected to depend on the fraction of the area devoted to  ${}^3\text{He}$  gas since more “exposed” molecules give better absorption. Detection efficiency is also dependent on the diffusive nature of the neutron since the nearby environment (e.g. supplied moderator, a body, even air) can allow neutrons leaving the detector volume to be scattered back into it, adding some additional detection fraction from neutrons detected on the bounce back. Without proof, the intrinsic efficiency should roughly follow the expression below:

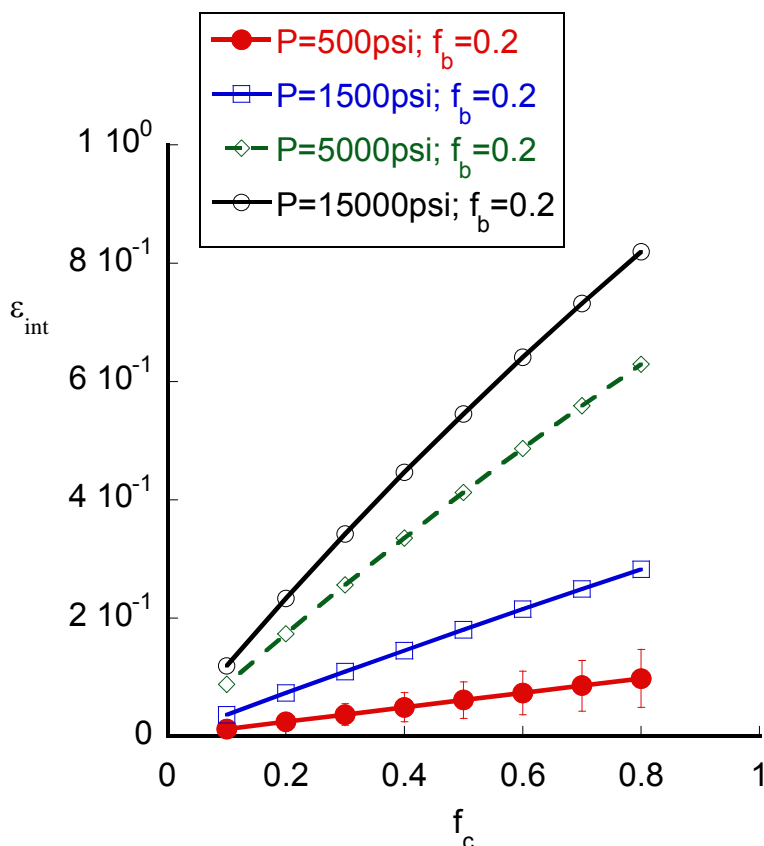
$$\begin{aligned} \varepsilon_{\text{int}} &= f_c \left( \frac{I}{I_0} \right) \left[ 1 + \sum_{n=1}^{\infty} \left( \left[ 1 - f_c \left( \frac{I}{I_0} \right) \right] f_b \right)^n \right] \\ &= \frac{f_c \left( \frac{I}{I_0} \right)}{1 - \left( 1 - f_c \left( \frac{I}{I_0} \right) \right) f_b} \end{aligned}$$

**Equation 2.2**

Here  $\varepsilon_{\text{int}}$  is the intrinsic efficiency,  $f_c$  is the fraction of device area which is capillary,  $f_b$  is the single bounce probability of a neutron passing the detector plane and  $I/I_0$  is the attenuation fraction for the thermal neutrons.  $I/I_0$  is the pressure dependent term. Figure 1 and Figure 2 illustrate the relationship between pressure, neutron albedo  $f_b$  and the areal fraction  $f_c$  of the detectors. For our current functional experimental device designs,  $f_c$  ranges from 0.1 to 0.8 as is discussed in the experimental plan for the safety work. The uncertainties caused by albedo ( $f_b$ ) are configuration dependent and vary over a nominal value of 10% for fast neutrons and up to about 80% for thermal. Current neutron detection experiments at pressures of 500 to 1500 psi of  ${}^3\text{He}$  and up to 800 psi of Xe are being conducted, but are not included in this safety study.

The uncertainties caused by albedo ( $f_b$ ) are going to be configuration dependent and vary over a nominal value of 10% for fast neutrons to about 80% for thermals. The figures illustrate how

these detectors are expected to compare when considering that current state-of-the-art devices are 15-25% efficient when embedded in moderator.<sup>1</sup> The large (15-25%) range is due to variations that may be observed in spectrum and shielding.

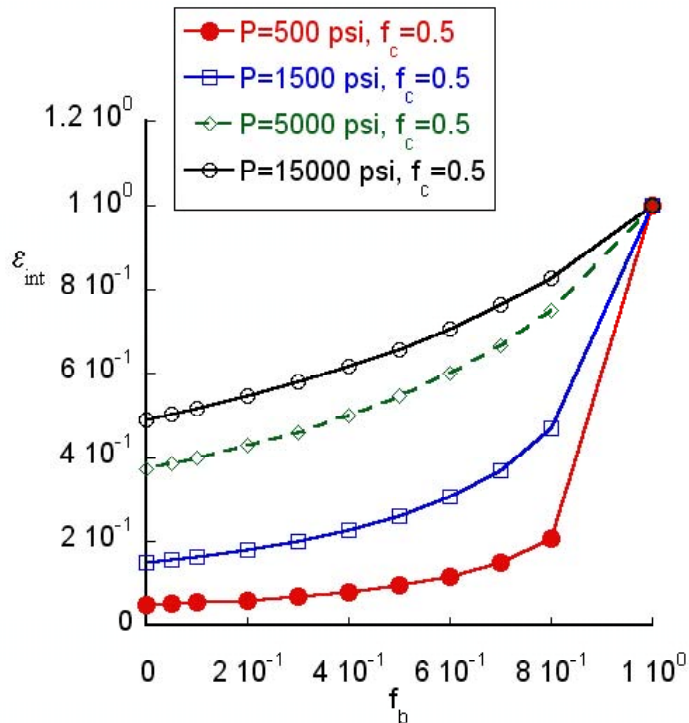


**Figure 1. Simple model estimate of efficiency as a function of capillary area fraction  $f_c$ . Individual curves show effect versus  $^3\text{He}$  pressure. The predicted design space has a maximum area fraction of about 80% at 1000 psi, falling to 50-70% at 5000 psi in FR4 or about 50% at 10,000 psi in a 0.7 mm thick Si wafer. Use of  $f_b=0.2$  allows for conservative estimates from this model.**

There are some counter intuitive issues associated with the graphs. For example, conventional tube arrays in moderator have only have a 20% efficiency. The tubes themselves are nearly opaque to thermal neutrons passing through the middle. Yet the opacity is strongly dependent upon neutron spectrum and most neutrons do not pass through the center but cross a much smaller effective diameter. Again the capillaries allow us to approach the condition of a uniform flat sheet of  $^3\text{He}$ . It is both this aspect of the geometry and a reduction in self-shielding (absorbed neutrons reduce the flux at the core – reducing the efficiency that neutrons ‘see’ the  $^3\text{He}$  nuclei) that result in the improved efficiencies. Another initially counter intuitive aspect of the design is that we expect a higher intrinsic efficiency than the volume fraction. There will always be losses in the inactive volume however the neutrons diffuse and some of them scatter back in- this is a core aspect of the simple model. The model accounts for backscattering. When a more sophisticated 3D code (in this case COG) is run, results show that the average thermal

<sup>1</sup> \*Reuter-Stokes Proportional tube, 4 atm  $^3\text{He}$  1” diameter embedded in moderator.

neutron crosses an embedded surface 4 times before being lost to the system. Therefore if your detector is highly absorbing for say 10% of the surface on the each of the next few bounces you still have that 10% chance of absorption (this only works for a finely divided surface). In a large surface (large with respect to the diffusion length  $\sim 1$  cm) that effect will not be observed. Therefore you will not see much of that effect with a large tube (for instance 2cm dia.) separated by a 1cm piece of moderator. This is one reason distributed capillaries are more efficient than arrays of tubes. In this sense the goal is really to have a sheet of  $^3\text{He}$  rather large discrete bundles of tubes.



**Figure 2. Dependence of expected intrinsic device efficiency using a fixed area fraction of  $f_c=0.5$  at various pressures. Neutron albedo,  $f_b$ , is typically about 15% for fast neutrons and 80% for thermal neutrons. Depending on configuration and source spectrum these numbers can vary. Mathematically the model goes above  $f_b=0.8$  however the model assumptions are expected to be inaccurate above that point. The point at  $f_b=1$  is shown merely for completeness.**

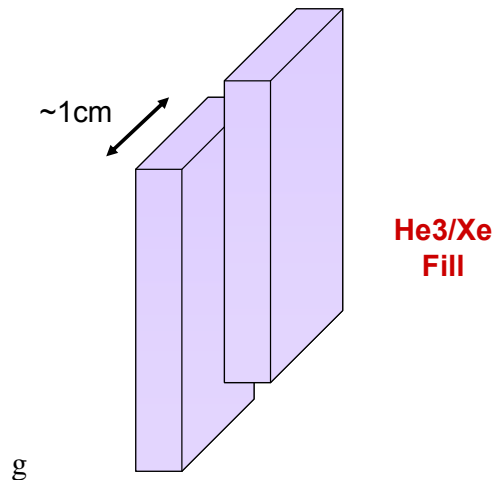
In its simplest form the strain limitations on the gas-containing wafer is expected to follow  $\sigma=P/A$  where  $s$  is the stress caused by pressure,  $P$ , in the capillaries and  $A=1-f_c$ .

Rough estimates of detector intrinsic efficiency per unit area can be calculated as a function of  $f_c$  (area fraction) which is capillary and partial pressure of  $^3\text{He}$ . Due to sensitivity to neutron diffusive effects and source spectrum, these numbers are approximate. They are on the low-side of what is expected. For comparison purposes the current designs of macroscopic  $^3\text{He}$  tubes combined with moderator have experimentally determined and modeled efficiencies of 15-25%.

The simple efficiency model has limits in that thermalization and parasitic absorption are specific to local environments and materials. However, for most problems values for fast neutron albedo of ~15% and thermal neutron albedo of ~80% are typically reasonable. This places bounds on expected performance. For the range of accessible pressures, efficiencies of more than 40% are expected and this includes geometrical losses and other system losses.

It is this high intrinsic thermal neutron detection efficiency combined with the high potential for large area mass-producible detectors that makes this such a competitive and compelling neutron detector. Efficiencies of 0.15 to 0.6 are expected via this simple model as compared to 0.15 for existing devices. More sophisticated radiation transport models which account for the full neutron spectrum suggest 0.7 or greater efficiencies as compared to 0.15 - 0.25 for existing  $^3\text{He}$  tubes.

The intent is then to have high pressure capillaries or channels in an array as shown in Figure 3.



**Figure 3. Schematic drawing of high efficiency capillaries arranged in a simple array.**

Large arrays of these capillaries or channels can be made within sheet-like materials or as modules using many different fabrication technologies. This is a primary goal of the development program. Photos of some of the FR4 devices are shown in Figure 4. The photos illustrate the configuration as assembled for testing.



**Figure 4. a) Backside photo of 9-plex FR4 array showing gas fill capillaries and Manifold, b) Front side photo of same FR4 array. No electronic connections are shown since tests were done without devices energized.**

The intent is to make these devices in large area arrays, in designs which are unobtrusive and have low-overhead requirements for field use. Safely-implementable devices of this type will also exhibit lower overall burdens and costs, with dramatically enhanced performance over existing technology. Again, this study is to assess the long-term potential for these proposed devices to be safe, because it is clear they would have significant advantages to the warfighter.

In summary, there is no possible way to get 2-3 times as efficient as a standard tube by merely going to high pressure. It is a combination of improved geometry and reduced self-shielding of  $^3\text{He}$  in the core that result in efficiency improvements. Note a secondary effect of high practical import: an improvement in efficiency of even a factor of 2 would result in effectively doubling the amount of  $^3\text{He}$  available making this limited resource go much farther.

### 3 METHODOLOGY

The proposed detectors will be filled with inert gas (He, Xe, or other noble gas), eliminating several common general safety concerns including chemical, fire, and explosive. Although these gases may be considered asphyxiates, which is typical of most gases, fielded operations involve very small total system volumes and are not intended to be done in closed areas.

It is the explosive and projectile issues that dominate this safety study, hypothetically including smaller-scale explosion hazards such as shrapnel generation, as well as bulk hazards presented by the potential energy contained in entire assemblies. The associated risks to personnel or equipment are shrapnel and device fracture. It should be noted that the loss of expensive  $^3\text{He}$  gas, at current costs of approx. 140\$/liter at STP, is also highly undesirable.

There are three primary elements to this safety analysis. These are the analytical comparison section, numerical modeling via finite element analysis, and test & experimental. When considering the safety of a pressurized system, context is useful. For this reason, the safety aspects of the devices in this study are often discussed in terms of relative concern with respect

to widely used items. The analytical section is used to qualify the relative concerns among familiar technologies. The theoretical analysis allows us to scale current designs to those tested and gives context for potential design space. Lastly, the experimental section contains observations and results that may be used to improve devices.

Pertaining especially to DOT guidelines and regulations, the factors that affect safety for pressure containers are total stored energy, energy per unit area and the time of release of the energy. The simplest and most direct method to compare relative safety of these devices is to know the absolute pressure hazard and compare it to the stored energy and energy density, analytically. Additionally, finite element analysis of static devices under pressure and a complete technical analysis, including deliberately insulting devices with a projectile, were performed.

Finally, it should be noted that drop tests pursuant to military guidelines were initially discussed. However, similar information involving fewer variables and requiring less specialized equipment and resources was obtainable with projectile impact tests.

### **3.1 Safety Comparison of Proposed Devices to Existing Technology**

The reason for this study is to put the potential safety of these devices in perspective. One way perspective is gained is by considering a number of common threats and conditions with respect to each other, as shown in Table 1. In the table, aspects of the safety threat including pressure, amount of stored energy, stored energy per unit area, and possible energy rate of release, can be compared to the consequence observed.

It is important to clarify energy per unit volume is important because it can give you the total energy released in a catastrophic failure. However, energy per unit area is what matters in the context of a surface explosion like this. The total stored energy for this design is not as relevant as the energy per unit area. Since pressure is released quickly, energy per unit area represents what would cause damage. Therefore we quote energy per unit area as the more important figure of relative merit.

The most significant correlation between consequence and the metrics in the table is the rate of release of the energy and the amount of stored energy. This combination best characterizes the major safety concern. Other key observations include the following:

- All other threats in Table 1 are transported routinely by DOT and considered manageable.
- Consequences from the energy release from an individual capillary are negligible. The projectile itself does far more damage than decompression of the capillaries.
- The projectile has far more energy present than the stored energy in the capillaries. Only if sympathetic failure occurs [where one capillary failure triggers another], is the energy release in the failure of a device able to add appreciably to the overall consequence set based on energy release.
- The distributed nature of large arrays of these capillaries is what makes them inherently safer than large discrete tubes.
- 

The interpretation of this analysis is that the bulk explosive hazard of an array of capillaries like that contained in the proposed devices or device arrays is minimal.



**Table 1. Comparison of relative safety concern for various common threats.**

Safety Threat	Metric					Consequence/Comment
	Pressure (psi)	Energy (J)	Energy/Volume (J/cm <sup>2</sup> )	Time of Release	Energy/Time (J/s)	
10 gal. Gasoline tank, 1m <sup>2</sup> tank	~0	2.5 x 10 <sup>8</sup>	2.5 x 10 <sup>4</sup>	Burn - Hours	70	Major consequence of fire and explosion
				Vaporize and Explode – 10 ms	2.5 x 10 <sup>6</sup> Explode	Routine hazard highly tolerated
3 g, 500 ft/s projectile	na	40	1.8	Millisecond	1.8 x 10 <sup>3</sup>	Moderate bullet speed, kill one person
Array of 4 <sup>3</sup> He tubes @ 10 atm, 1”dia. x 24 in. long (100in <sup>^2</sup> )	150	1200	8	Rupture (ms) Leak	8 x 10 <sup>3</sup>	Knock someone down; more energy than the projectile but much larger area
1 cm long, 300μm x 300μm capillary at 10,000 psi	10,000	0.006	0.2	Millisecond	20	No large area damage, small, fast low mass projectiles may be created.
100 cm <sup>2</sup> area panel; 1667 capillaries*	10,000	10	0.1	All in 1 ms	100	-If entire module failed at once – consequences would be minimal - If as consistent w/experiments, lose 1 cm <sup>2</sup> and confined there would be no shrapnel
				Consistent w/ Experiments	1	
Scuba Tank @ 3000psi and 12L	3000	1.2x10 <sup>6</sup> Joules!	na	Rupture (10ms)	10 <sup>7</sup>	Destroy a room or car trunk
				Slow Leak/ Crack (1 hr.)	3x10 <sup>4</sup>	Or, boil a gallon of H <sub>2</sub> O based on the energy release

\* The Sandia design SMART system array of four large tubes embedded in thick polymer is of nominally 0.2 m<sup>2</sup>. This design is expected to be 1-1.5 times as sensitive as the larger array.

Close inspection of the columns and rows of Table 1 shows it is very difficult to enter a hazard regime with large amounts of stored energy or fast release of energy. Hypothetically, there are clearly two times when the proposed devices potentially become a hazard. First, if one device sets off another, which we call parasitic or sympathetic failure. The worst case situation would be a release of nominally  $(10 \text{ J}/100\text{cm}^2) \times (\text{area of failed devices})$ . Second, the creation of small fast moving shards that could cut or tear might occur. The following experimental components of this report show that both hazards can be decisively dealt with.

Although this analysis indicates the bulk failure of the devices is not an explosive concern there are some special circumstances that need to be considered. Specifically, high velocity shrapnel and shards are hazardous but very difficult to computer model. It is beyond the state of the art to model explosive fracture in a deterministic way. However, relatively inexpensively, prototypical devices can be observed during overpressure failure and impact. These observations during failure have been done, with the results discussed below.

It should be noted that for the ductile FR4 devices the shard situation was never encountered. For the brittle Si devices a layer of fibrous duct tape stopped all particulate debris.

## **3.2 Finite Element Analysis of Capillaries**

Static finite element analysis modeling (FEM) of devices using ANSYS was done to understand limitations on design in terms of yield stress. Some devices were also modeled in ProE Mechanical with von Mises yield criteria. While stress criteria models do not provide a complete understanding of how the devices will fail or whether sympathetic failure will occur, it does provide information indicative of materials and thicknesses required to avoid unacceptable yields. The static models are very useful in determining initial design boundaries and expected performance.

It should be noted that failure mode analysis of direct and sympathetic failure requires dynamic modeling beyond the scope and resources of this work. However, projectile and burst tests are more than adequate substitutes, providing a more realistic and complete understanding of failure modes.

### **3.2.1 FR4 Devices**

FR4 material was not modeled or FEA-evaluated since the design used for FR4 devices incorporates a compressed gasket, for which standard engineering calculations, fastener spacing, material allotment, and dimensioning were deemed acceptable for use at approximately 4000 psi. Additional detailed discussion of FR4 devices is contained in section 6.3.

### 3.2.2 Silicon Devices

The silicon designs were spiral, ladder or porous bed concepts. These different design types result in different electrode shapes and different channel cross-sections. The differences in channel cross section affect how the channel holds stress and how much gas pressure the channel can withstand. Corners can be filleted (rounded) to provide stress relief. The differences would also affect how the electrodes would be structured. For stress purposes the spiral and ladder designs are similar in that there is a nominally square cross-section and the channels are simply filled with gas through a manifold. The porous bed designs employ Bosch etched through holes created in a central wafer with wafers containing electrodes bonded on each end. The porous bed designs may be stronger because of reduced stress concentration effects at the corners. Unfortunately because of the bonding problems these designs were not tested to high pressure.

The modeling shown here is for anodically bonded 0.7 mm thick Si wafers. Early tests showed that the strength of the bond is the same as the bulk material. This meant that the bulk material failed before the bonded wafers delaminated. The modeling is done in 3Dimensional structures using the initial CAD drawing for the lithographically processed material. Bulk material properties were applied across the joint.

In this report we only show the most important features from the modeling that has been performed. Figure 5 shows a characteristic plot of the stress within a spiral configuration. The peak stress is 227 MPa. Failure occurs at 5 GPa in Si. This indicates that there is a 20x safety factor. Because there are stress concentration points shown at the channel edges, we performed high resolution modeling in two dimensions to better capture the effects of stress concentration at the joint. Figure 6 shows the effect of stress concentration. In the higher resolution model of the corner the maximum stress is slightly below 600 MPa. There is an apparent reduction in safety factor due to the presence of the corner. The implication is that attention must be paid to the corner radius during manufacture. Even at this nominal 5 micron radius (used in the model) the value of the peak stress implies a safety factor of 8x. The porous bed design should exceed these values.

The modeling results indicate that the Si designs should function at pressures up to 10,000 psi with 300 micron x 300 micron channels when made in 675 micron thick Silicon wafers.

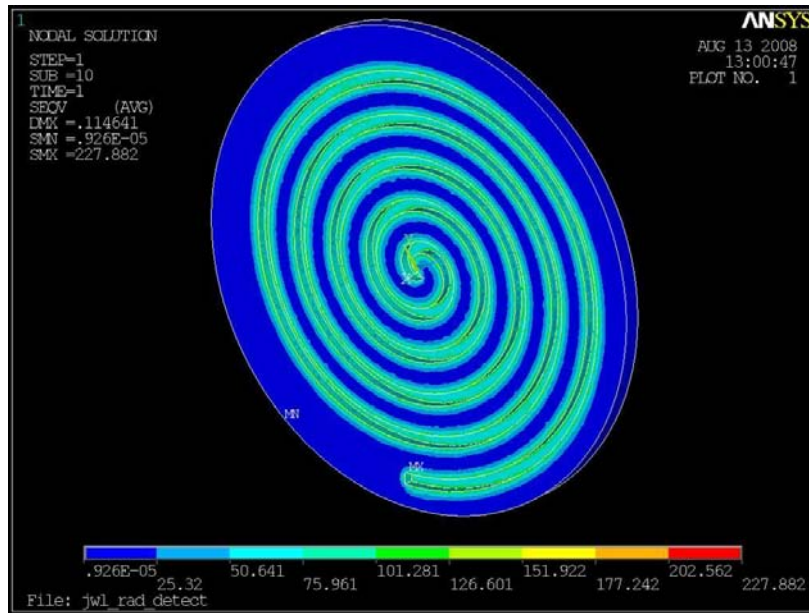


Figure 5. 3D image of stress versus location in ANSYS model of spiral capillary embedded in Si wafer. 10,000 psi of gas fills the capillary.

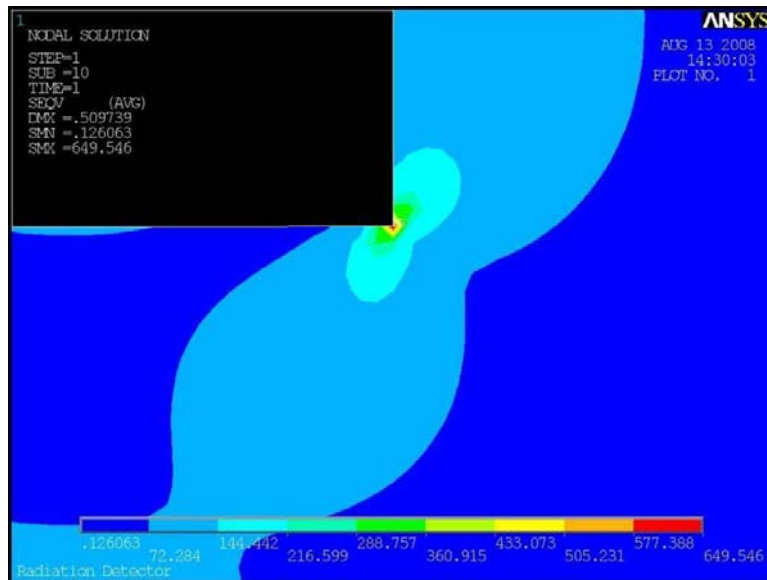


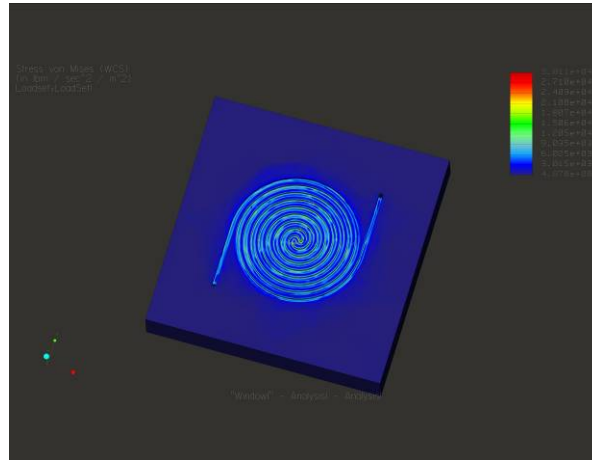
Figure 6. 2D model of Si channel. This cross section image provides a high resolution look at the stress in the corners.

### 3.2.3 Parmax devices

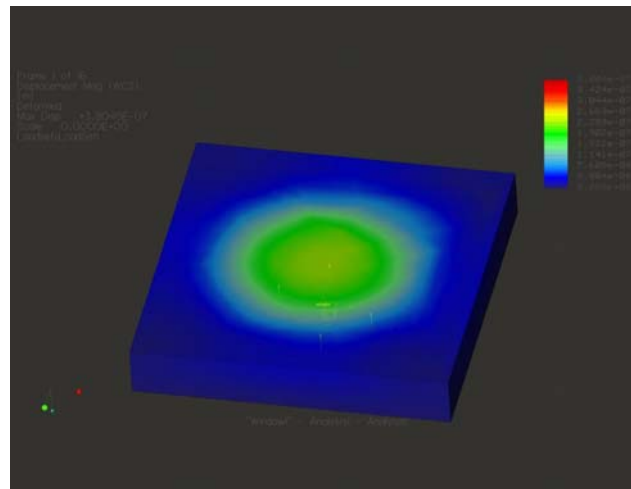
Calculations were done for the Parmax devices assuming a uniform diffusion bond was created. Diffusion bonds should give bonded areas the same failure strength as the bulk material. More specifically, calculations suggested Parmax devices should be able to exceed a safety factor of 4 in 0.125" thick wafer substrates with channels pressurized to 5000 psi. However, experimental

evidence to date indicates that this material cannot be adequately diffusion bonded without additional development.

Most importantly, experimental results indicate that upon impact these devices shatter in an undesirable way. For these reasons we are no longer pursuing this material, but we include the modeling for completeness, documentation, and in anticipation of possible future successful diffusion bonding methods. Results shown here are for .0625" thick wafers and channel pressures of 4000 psi, with material allotments providing a safety factor of 4. Stress profiles of the channel-containing base wafers are shown in Figure 7 and Figure 8.



**Figure 7. Stress distribution around a spiral channel machined into Parmax device base wafer.**



**Figure 8. Stress distribution on overlay of Parmax device.**

### 3.3 Projectile and Burst Tests

The goal of projectile and burst tests is to qualify and quantify the hazards presented by various device configurations when pressurized with gas and impacted by a projectile with specifically selected kinetic energy. As denoted in Table 1 and discussed in section 3.1, this amount of energy represents a slow rifle round, for instance, fired from a distance, or after penetrating protective shielding. This energy is also ideal for device material tests, since it results in penetration of the device by the projectile while also straining the overall device and means of attachment. As stated in section 3.2, these tests are real-world substitutes for dynamic modeling, and are also likely to be considerably less expensive.

Failures unforeseen by static models include two general expected failure types. These are 1) “impact zone” failure of only channels at or very near the impact zone, or “sympathetic” failure, of channels near the impact zone and 2) additional failures in areas or devices relatively far from the impact zone. An additional but unexpected type of sympathetic failure includes possible stress propagation from one device to another resulting from the method and type of assembly used to arrange devices into larger arrays, rather than from the release and propagation of potential energy from He in the channels. For instance, stress on the overall device during impact may induce stress concentrations at attachment points that may cause fracture or yield at those locations.

It is likely that the fractional area of the channels contained in a device is a primary factor predictive of sympathetic failure, i.e., more fractional area covered by channels means fractionally less material between channels, which provides less structural support for the same levels of material stress (assuming channel pressures are constant as fractional area varies). Shape-related parameters, for instance serpentine, spiral, or otherwise-configured channels, are also possibly important, but are likely secondary considerations compared to fractional channel area, especially if a projectile of the appropriate scale is selected, as discussed further below.

To adequately test for the presence of both failure types while destroying fewer target devices, a single projectile relatively large compared to channel and between-channel dimensions, but small enough to cause direct damage to only one device and its associated channels, was chosen. A projectile of this scale, and of a kinetic energy expected to result in penetration or shattering of the target device, should induce stresses at both the channel and between-channel scale as well as on the scale of the entire target device.

Projectile shape factor and rotational characteristics are thought to be less significant, and beyond using a bullet-nosed projectile for aerodynamic stability and target accuracy, no attempts to control or quantify those was thought necessary. With adequate recording instruments and methods, choosing a projectile of the aforementioned size maximizes the probability of distinguishing between direct and sympathetic failures.

The recording instrumentation includes a digital camera with extremely high imaging rates capable of distinguishing between impact zone and sympathetic effects, a microphone to capture the time history of escaping gas from device channels, a pressure transducer and data logger to monitor channel pressure, and a primary trigger enabling sequencing of events (Figure 9).

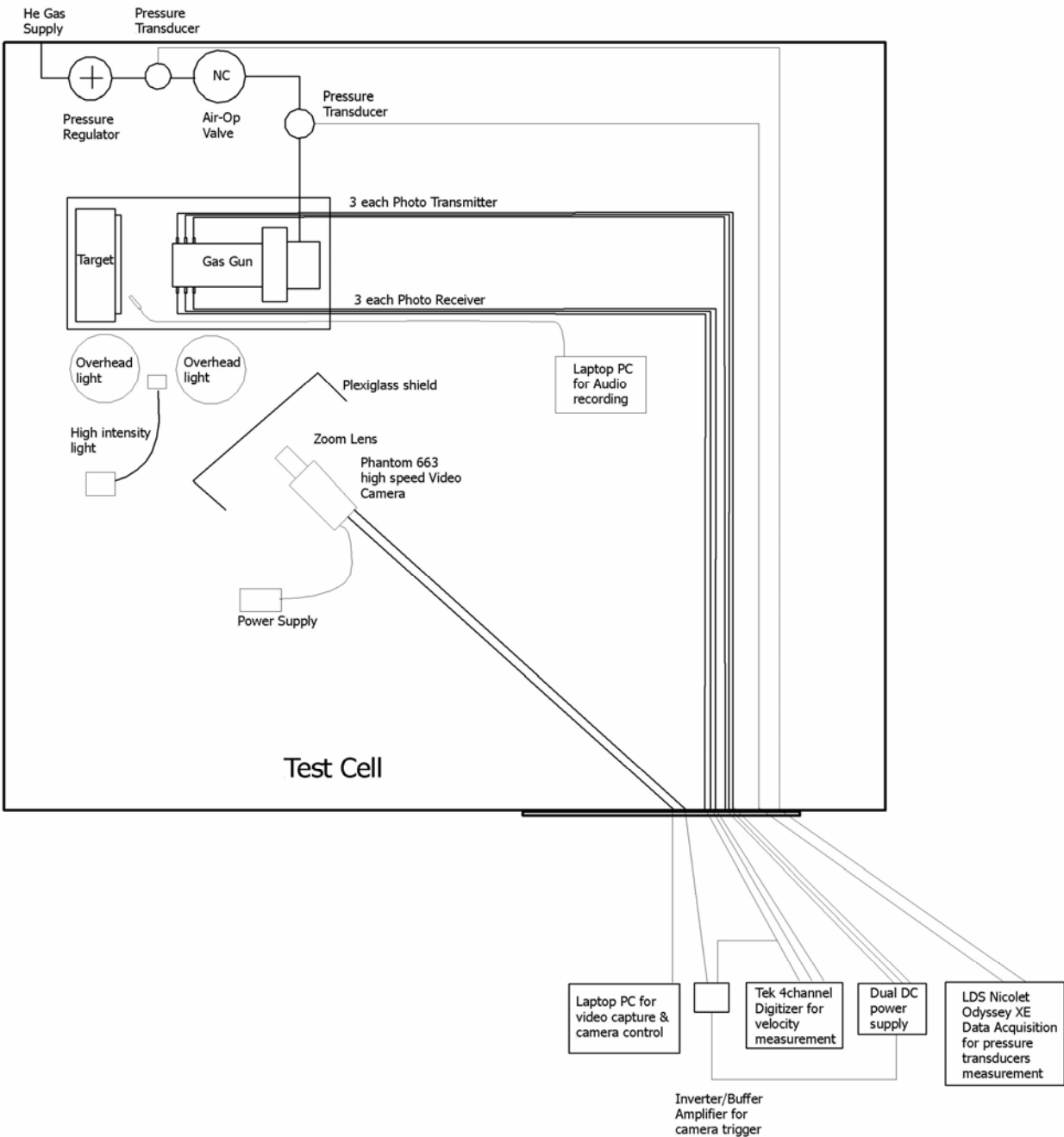


Figure 9. Projectile Impact Test Set-up.

## 3.4 Experimental

### 3.4.1 Facilities

Projectile impact and burst tests were conducted in a high pressure test facility containing explosion-rated cells equipped with autonomous instrumentation, controls, data loggers, gas

valves, gas projectile-generating equipment, and a high-speed camera. All equipment is housed within the cell, which is unmanned during testing (see Figure 10). Projectile impact tests were chosen in place of drop tests, since drop tests would have introduced more variables and specialized equipment requirements in order to obtain similar data quality.



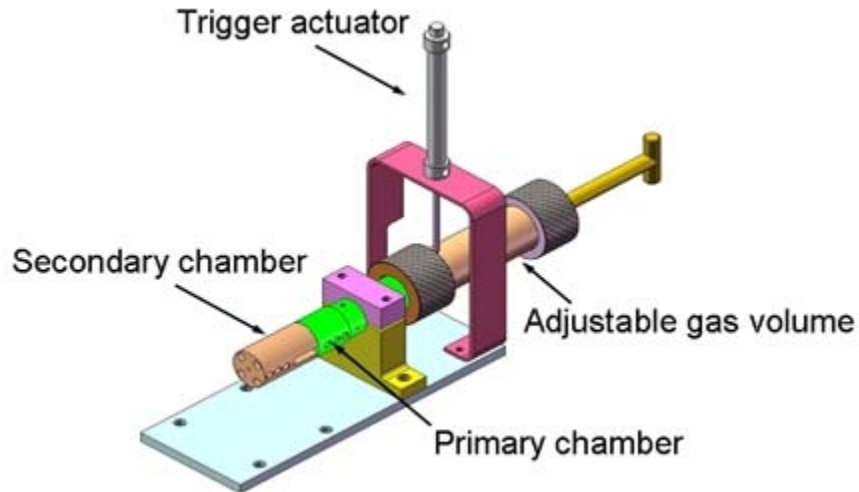
**Figure 10. a)High pressure test lab, SNL Livermore, b)Overview of projectile impact test set-up.**

### 3.4.2 Set-up Description & Detail

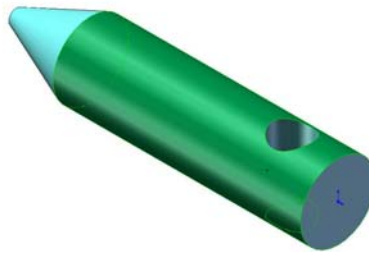
The experimental set-up generally includes three categories, namely, impacting projectile, target devices impacted, or imaging & data acquisition equipment. For all devices and all tests, the same impacting projectiles were used. These were 3.5 gram bullet-nosed titanium slugs ejected from a custom gas-driven gun with velocities of 200 m/s, with a total kinetic energy of approximately 50 Joules. A nominal 10% variation is observed between events.

The projectile generator shown in Figure 11 uses helium gas to force a cylindrical projectile down the primary barrel and out through the muzzle end. Projectile velocity is measured at the send/receive optical sensors mounted in pairs at six positions on the secondary barrel. The volume of the pressure chamber is adjustable with a plug and adjustment screw. Although a variety of different test projectiles can be used, the basic “bullet” concept is the foundation for design and is illustrated in Figure 12.





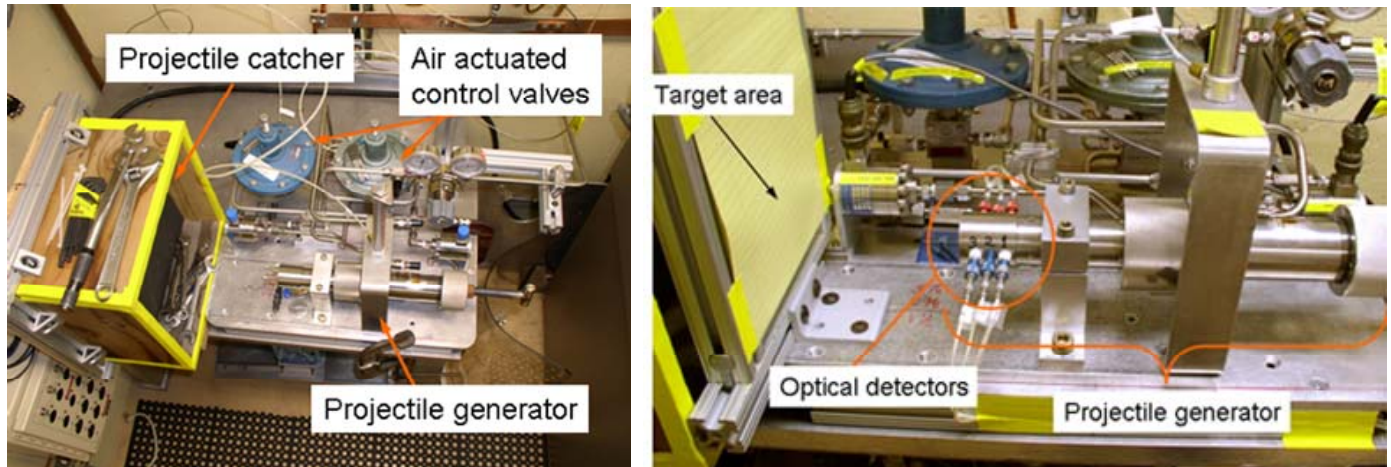
**Figure 11. CAD rendering of projectile generator.**



**Figure 12. Projectile with retaining pin cross hole 1. Projectile weighs 3.5 g.**

The approximately 1/8" diameter thru-bore in the test projectile is mated to another bore on the primary barrel and then locked into alignment by a solid pin connected to an air-driven cylindrical actuator. To initiate firing the actuator moves the solid pin laterally to release the test projectile.

With the projectile unrestrained, there are unbalanced forces on a rupture disk behind the projectile. High pressure in the pressure chamber ruptures the center of the disk, enabling expanding gas to propel the test projectile forward and down the bore of the primary barrel at high speed. Ejected projectiles are caught in a "catcher" constructed of multiple layers of materials such as wood and polymer that absorb kinetic energy (Figure 13).



**Figure 13. a) Projectile gun system & catcher, b) Close up of projectile generator.**

### 3.4.3 Target Devices and Arrayed Assemblies

All devices are assembled from 1/8" base wafer and 1/8" coverlay w/ via holes, although bonding methods differ amongst devices. Target device materials of construction were silicon, Parmax, or FR4. Cover layer and base wafers were sealed in a variety of ways, depending on the wafer material. For Parmax assemblies, Scotchweld DP420 2-part epoxy was used to bond coverlayer-to-base, since diffusion bonds did not work well for that material. Silicon devices were diffusion bonded using standard well-documented techniques. FR4 devices used compressed custom PEEK gaskets to seal between wafers. All of the devices were assembled with nearly identical high pressure gas connector configurations, including 365 x 275 mm stainless steel tubing between the helium source and PEEK plenum manifold, and from the plenum to each discrete device (referenced in Figure 4a).

Devices were arranged over a 200 mm<sup>2</sup> area in 3 X 3 device arrays in preparation for impact tests. See Figure 4 for front and backside images of assembly. In all but one of the tests where a solitary FR4 device using a bolt-down attachment method was tested, devices were attached to an aluminum-skinned, 1/2" thick, 6" x 6" section of honeycomb backing material, with 1 mm thick double-backed Scotch brand adhesive tape, with an approximately 0.5 mm gap between devices. The honeycomb backing is the expected material to be used in large array assemblies. The double-backed sticky tape was chosen as a convenient means of attachment that does not pre-stress the devices, although it is not intended to be used in final fielded assemblies.

### 3.4.4 Data acquisition equipment

Several data acquisition components are shown in Figure 9 and Figure 11. A manually operated instrumented control panel external to the test cell was used to operate the projectile generator.

Before exiting the muzzle, the projectile traverses the barrel of the generator, passing optical interrupt photodiodes in the secondary barrel that provide projectile velocity data. Optical

interrupts also provide a trigger signal that initiates a 1.5 second duration data acquisition window.

Data acquisition includes images captured by a Phantom 663 high speed camera operating at 6600 frames per second, and pressure data recorded by an LDS Nicolet Odyssey XE Data Acquisition system. The Odyssey monitors pressure transducer signals associated with both projectile driving pressure and device channel pressure. A separate laptop is used for electronic control and parameter manipulation for the Phantom 663 camera.

## 4 DOT REQUIREMENTS

**(49CFR179-- Subpart B\_General Design Requirements).** Inspection of these DOT regulations showed that this specific set of technologies is one with which the DOT is unfamiliar. Specific regulations and requirements such as General Design requirements, relief valves, etc., generically apply but could use modification, or perhaps addition of a subsection for inclusion of these types of pressure vessels (i.e., miniature arrays of high pressure capillaries and/or channels).

**(49CFR178.75-- Sec. 178.75 Specifications for MEGCs).** This CFR-comes closest to describing our proposed devices, specifically in the sections pertaining to MEGC (multiple element gas container regulations; 2007). Comparison of the types of technology for which this CFR developed appears to have Scuba tanks or similar scale devices in mind when the code was written and should be feasible under this CFR certification.

**[49CFR192-- Subpart A\_General]** Certain specific issues, such as pipeline and hazardous material aspects of the code provide no reason to include our technology. Materials anticipated for use with our technology are primarily noble gases and nonhazardous confining substrate devices (i.e. FR4).

**[49CFR172.102-- Sec. 172.102 Special provisions]** Preliminary inspection of the special provisions section suggests no concern exists for inert/noble gases, and there are no additional issues in this area.

**[2007 49CFR173.306-- Sec. 173.306]** Specific vessel and air shipping of limited quantities of compressed gases are covered by these CFRs. Based on the included regulations formula, our proposed device does not exceed the requirements (20,000 psi). Clearly the regulations do not cover our particular type of design and special consideration may need to be made.

**[49CFR171.8-- Sec. 171.8 Definitions and abbreviations].** The closest example conceptually was a bundle of cylinders not to exceed 3000 L of water. Our proposed device clearly does not exceed this requirement, but once again the CFR did not have our proposed technology in mind.

[49CFR176-- **Subpart G**] Galvanic action, aging, pressure cycling , of containers not more than 1000L, including Detailed Requirements for Class 1 (Explosive) Materials also needs to be considered but no modification should be necessary.

[2007 49CFR180-- **PART 180 - CONTINUING QUALIFICATION AND MAINTENANCE OF PACKAGINGS**] The correct contact needs to be identified, and work towards modifications regarding lifetime issues with the capillary devices needs to be done.

[49CFR180.407-- **Sec. 180.407 Requirements for test and inspection of specification**]

Existing inspection requirements can be met, but requirements should be developed which are specific to the proposed technology. The most current guidance is SP11650\_dot specs.pdf, attached as an appendix, and this work does allow systems at operating pressures up to 8500 psi.

Our conclusion after consideration of the aforementioned CRFs is that with DOT consultation, a common understanding of our proposed technology should result. Also, it is reasonable to assume that the special circumstances presented by our proposed technology can be addressed by existing CFRs and allow for routine shipment.

## **5 RESULTS, DISCUSSION AND CONCLUSIONS**

### **5.1 General Overview & Common Results**

Discussion and conclusions are presented according to device material of construction. These procedures involved impact tests of devices with unpressurized channels, devices with channels pressurized up to 4000 psi prior to impact, and devices with reinforced incident faces. Additionally, burst tests where no impacting projectiles were used, but devices were pressurized to the point of failure, were performed.

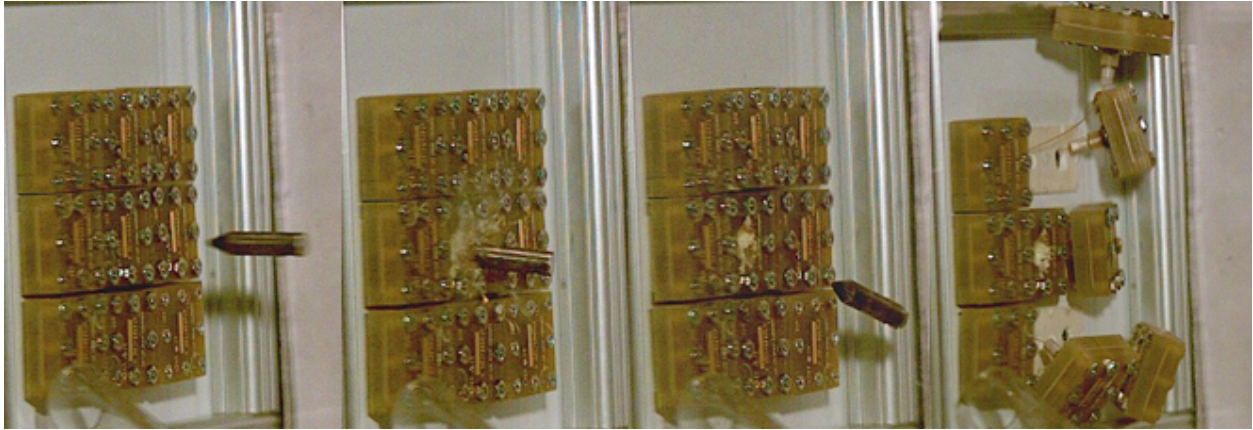
In all tests, no other devices other than the target device within an array was damaged by impact, but many devices would detach from the double-stick tape used to adhere them to the aluminum-skinned honeycomb backing plate due to reflexive action, or recoil, of the backing plate after impact. This was not the case with the arrayed FR4 devices that were bolted to the backing plate, i.e., all arrayed devices remained attached after impact.

For all devices tested, comparing pressurized and unpressurized post-impact devices showed no difference in failure mode or characteristics, supporting the conclusion that if potential energy of the compressed gas is small relative to projectile kinetic energy, damage is overwhelmingly caused by the projectile. However, the type and severity of damage varies considerably depending on device material of construction and is described as follows.

### **5.2 FR4**

FR4 devices exhibited ductile failure during impacts, with material yielding at the impact zone without crack propagation through nearby material. The impacting projectile did not completely pierce the target device, and some material at the impact zone thermally decomposed as

indicated by generation of a small amount of smoke and fumes during impact (Figure 14 and Figure 15).



**Figure 14. Successive images of projectile impacting array of 9 FR4 unpressurized devices. Simple double sided tape attached the devices to the support.**

Since these devices did not generate fragments, no reinforcement with duct tape or otherwise was needed. The next test was an array of FR4 devices bolted down to the honeycomb backing plate and pressurized. The intent of this second test was to determine if pre-stress induced by the method of assembly, or stress propagation from impact through the assembly as well as pressurization, would result in failures not previously seen.



**Figure 15. Successive images of projectile impacting array of nine FR4 pressurized devices. Devices were bolted to the support plate.**

Results were favorable, with only the target device showing any damage from impacts, and without adjacent devices detaching or losing pressure. The target device released the internal pressurized gas within the detectors. Detectors which were not directly impacted held pressure upon later inspection. If capillaries would have been fluidically disconnected we suspect they would have continued to hold gas. The effect of the high pressure gas was 'negligible' based on the comparison of the non-pressurized and pressurized testing. Clearly the kinetic energy of the projectile dominated the event and the effect of the high pressure gas was negligible.

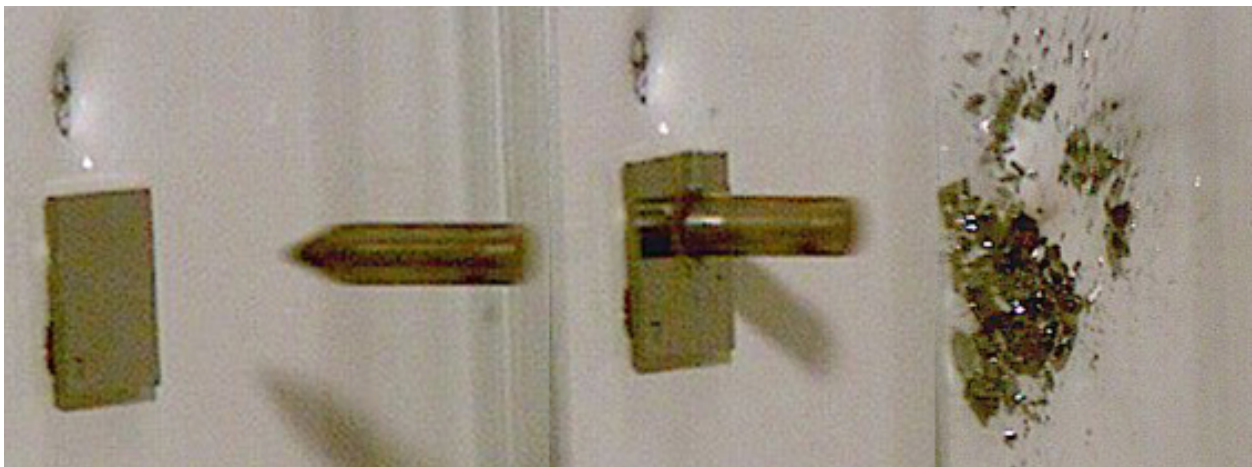
Burst testing of the FR4 configuration developed slow leaks at 4200 psi, then dropping to and resealing at 3300 psi. It could then subsequently be pressurized back up to 4200 psi, redeveloping leaks and resealing at 3300 psi. This pressurization-leak-reseal cycle was repeated numerous times and showed no signs of variation. With the slow leaks developing at 4200 psi, the FR4 devices were not taken to burst pressure. At some point, plastic deformation may prevent the re-sealing. This effect was not observed and we are currently unable to provide a good safety factor for an operating pressure.

The implication of limiting operation to 4000 psi is that we may need to move to a two-sided design in order to maintain maximum efficiency. This is not considered a difficult or problematic redesign. Caveats to this are that these conditions would not provide a good enough safety factor. Long term exposure at this pressure would likely cause eventual leaking, given the fact that it does not re-seal until 3300. Additional work and perhaps seal design will need to be done in order to meet our long-term goals. In summary, with the benefit of well-known mass production techniques such as multilayer overlapping and the ability to directly incorporate integrated electronics, since this is what this material is typically used for, FR4 is a strong candidate material for device manufacture.

### 5.3 Silicon

#### *Projectile testing of unpressurized Si devices for brittle failure analysis*

Silicon exhibited classic brittle failure during projectile impacts, shattering into small pieces all less than 4 mm<sup>2</sup>, with most fragments no larger than 1 mm<sup>2</sup>. Adjacent devices were minimally damaged, with small chips along edges nearest the target device, but with no other visible damage (Figure 16 and Figure 17).



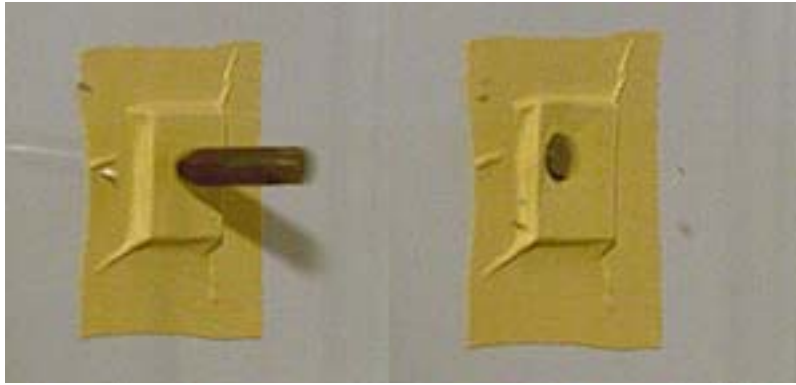
**Figure 16. Successive images of projectile impacting array of nine 2x2 cm silicon devices.**



**Figure 17. Successive images of projectile impacting array of 9 2x2 silicon devices.**

The projectile completely pierced the target device and continued on through the honeycomb backing plate, stopping only after penetrating two layers of 2" thick foam backstop of two different hardnesses, then impacting and insulating a high density Romel polymer plate. This additional travel through multiple backing layers confirms that relatively little projectile kinetic energy is absorbed by the Si target device. This may be a very useful result should energy propagation to adjacent arrayed devices need to be kept to a minimum, although all tests of all materials do not indicate that this is a problem.

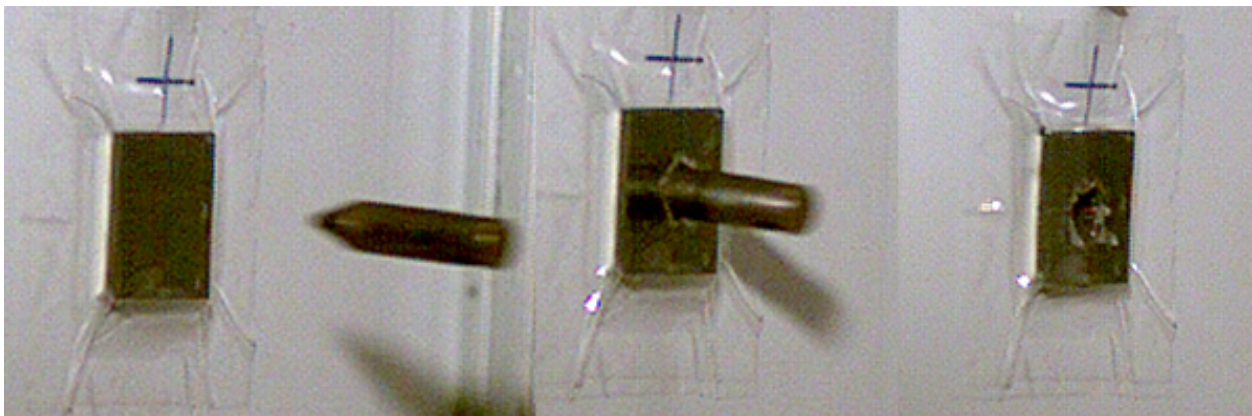
When the incident face of a solitary Si target device was reinforced with commercially available duct tape, carbon fiber weave, or cellophane tape, brittle failure was nearly eliminated, with far fewer and larger fragments generated, and with few fragments in the penetration path void left after the projectile traversed the device (**Figure 18**).



(a)



(b)



**Figure 18. a) Successive images of projectile impacting silicon device with yellow duct tape, b) Successive images of projectile impacting silicon device with carbon fiber weave, c) Successive images of projectile impacting silicon device with clear tape reinforcement.**

This is consistent with well-known mechanical stress reduction techniques whereby stress concentration in ductile layers overlying brittle materials greatly reduces the stress and strain in



underlying materials. An example of this are Polymicro glass capillaries, which, when coated with a thin polyamide layer, can bend with very small radii. Such reinforcement strategies may be used for future designs involving fragile devices or device assemblies made of Si or other brittle materials, or if future devices or arrayed assemblies might encounter extreme conditions.

*Impact testing of Si devices for sympathetic failure were not conducted.*

The most disappointing aspect of these tests was that in the manufacture of the Si-bonded wafers a mistake was made and the bonding was not able to withstand pressures above 20 psi. These weak bonds were determined to be caused by an inadvertent change made in the bonding methodology. The bonding was performed with a 1 hr process time; the initial devices were made with a 9 hour bond. We do not know why they only processed them for 1 hour. The poor bond was not discovered in time to make new wafers and include different wafers in the experiment. This was also mentioned in Section 3.2.2.

*Si testing summary*

Shrapnel from impacted unpressurized Si devices is easily contained. We believe that the relatively benign impacts (Fig. 18) combined with the relatively low ratio of the kinetic energy/area of the gas as compared to the energy/area of the projectile will result in a safe design space for Si wafers, Pressurized sympathetic failure tests were not performed. Further testing needs to be done.

## **5.4 Parmax**

Parmax devices showed a combination of brittle and ductile failure, with impacts causing fewer, larger, and more violent crack propagation through the device material, and generating far fewer and larger fragments than with Silicon. The impacting projectiles frequently did not completely penetrate through the entire device, although the nose typically partially exited the base wafer, and pierced the aluminum skin of the honeycomb backing plate (**Figure 19**).



**Figure 19. Successive images of projectile impacting array of nine pressurized Parmax devices.**

Reinforcement identical to that with silicon devices did not contain generated fragments or shards. Additional tests with a Parmax device array bolted to the honeycomb backing plate, as were performed with the FR4 devices, were deemed unnecessary for a variety of reasons. Foremost of these were the generally poor impact tests results. More specifically, the relatively violent failure of these devices, in combination with the observation that impacting projectiles do not fully penetrate and exit the device, indicates that a relatively large amount of projectile energy is absorbed by the devices. This is not a favorable condition, i.e., the less energy absorbed by a device the better, since less energy is subsequently transmitted to pressurized channels, device electronics, and associated device subcomponents. By comparison, FR4 and silicon are far superior materials. We infer from the movie that sympathetic failure does occur in Parmax as fabricated.

In addition, Parmax devices have possible diffusion bond problems, since known solvent diffusion bond techniques did not work for this material. A preferred two-part adhesive that has worked well in past applications, including, for example, applications where injection ports bonded to device wafers have been tested to 10,000 psi without failure, was used instead to join base and coverlay wafers .

In burst tests with the Parmax devices joined by the aforementioned two-part epoxy, devices failed at the bond line at approximately 2000 psi. These devices could have gone to pressures of approximately 10 kpsi with dimensions and material allocations similar to silicon and FR4 devices.

However, experimental evidence obtained to date suggests this material cannot be diffusion bonded more ideally without additional development. Incomplete diffusion bonds likely caused the relatively violent shattering of Parmax devices observed upon projectile impacts.

Finally, integration of electronics is also problematic with Parmax, since solder traces cannot be directly applied to this material. For the aforementioned reasons we will not pursue Parmax further at this time.

## 5.5 General Conclusions

The experimental results, while consistent with the finite element analysis and relative energy calculations and considerations, are indisputable verification that the primary effects of impacts are caused by absorbed projectile kinetic energy, not stored energy in the channels. Photographic evidence is provided in the sections pertaining to specific materials above.

Acoustic results during projectile tests were most often indistinguishable from noise generated by impacts and subsequent reverberations. This was also generally the case for burst tests, i.e., noise generated during burst failure overwhelmed any acoustic indications prior to failure. However, in the case of the FR4 device, where repeated leaking and resealing occurred, acoustic monitoring helped verify the onset of both.

Since experimental impact and burst test results are consistent with analytical estimates of stored energy, energy density, and the energy and momentum characteristics of the projectile, and since FR4 devices did not exhibit sympathetic failure during either projectile impact, insult, or burst tests, it is confirmed that the high pressure gas contained within the channels is a negligible safety risk, up to pressures of at least 4000 psi. Silicon devices may prove acceptable with further testing. Parmax devices appear unacceptable and we suggest terminating development with this material.

Both the experimental work and modeling suggest that DOT requirements for FR4 and potentially Si devices can be met. Reconciling test results with DOT transportation guidelines will be discussed in the next section.

Since device designs fabricated from the aforementioned materials have been shown to be safe for personal handling, transport, and foreseeable fielded operation, including impacts, these detector systems have the potential to significantly impact neutron diagnostic systems used for ICF and for Non-Proliferation and Homeland Defense Applications.

## 6.0 Future Developments

Initially, we will be building 100 cm<sup>2</sup> device arrays, followed by 1 m<sup>2</sup> arrays. These arrays may be assembled from FR4 or Si-wafer devices; and, with either material, additional safety or structural failure testing will need to be performed. Both drop and impact testing will be performed as required by further stages of development and certification pursued to obtain special permit authorization from the US DOT.

**7 APPENDIX**

April 23, 2008

East Building, PHH-30

U.S. Department 1200 New Jersey Avenue S.E.  
of Transportation Washington, D.C. 20590  
Pipeline and Hazardous  
Materials Safety Administration

DOT-SP 11650  
(TWENTY-SECOND REVISION)

EXPIRATION DATE: November 30, 2009

(FOR RENEWAL, SEE 49 CFR ' 107.109)

1. GRANTEE: Autoliv ASP, Inc.  
Ogden, UT

2.

a. PURPOSE AND LIMITATIONS: This special permit authorizes the manufacture, marking, sale and use of non-DOT specification pressure vessels for use as components of automobile vehicle safety systems. The pressure vessels, charged with non-toxic, non-liquefied gases, are authorized for transportation in commerce subject to requirements and limitations specified herein. This special permit provides no relief from the Hazardous Materials Regulations (HMR) other than as specifically stated herein.

b. FIVE-YEAR TRANSPORTATION AUTHORIZATION: This special permit authorizes transportation of the pressure vessels identified herein for up to five years from the date of manufacture. This limitation does not apply to non-specification pressure vessels when installed in air bag modules. This special permit provides no certification of safety for end use environments and life cycles.

c. SPECIAL PERMIT SCOPE LIMITATIONS: This special permit only applies to a package when it is an article of commerce in transportation. The safety analyses performed in development of this special permit only considered the hazards and risks associated with transportation in commerce. The safety analyses did not consider the hazards and risks associated with consumer use, incorporation as a component of a vehicle or other device, or other uses not associated with transportation in commerce.

Continuation of DOT-SP 11650 (22nd Rev.)  
Page 2

April 23, 2008

3.

REGULATORY SYSTEM AFFECTED: 49 CFR Parts 106, 107 and 171-180.

4.

REGULATIONS FROM WHICH EXEMPTED: 49 CFR ' ' 173.301(a)(1), 173.302a(a) and 175.3 in that non-DOT specification cylinders are not authorized, except as specified herein.

5.

BASIS: This special permit is based on the application of Autoliv ASP, Inc. dated September 21, 2007, submitted in accordance with ' 107.105 and the public proceeding thereon.

6.

HAZARDOUS MATERIALS (49 CFR ' 172.101):

Hazardous Material Description

Proper Shipping Name Hazard

Class/

Division

Identification

Number

Packing

Group

Air bag inflators, or Air bag

modules, or Seat-belt

pretensioners

9 UN3268 III

Note: A safety system or component which contains a quantity of pyrotechnic materials must be classed and approved as provided for in ' 173.56 of the Hazardous Materials Regulations (HMR). If the pyrotechnic material augments the volume of the gas in the pressure vessel, or in any way enhances the performance of the compressed gas, the device must be tested in the same configuration as when shipped.

7.

SAFETY CONTROL MEASURES:

a. PACKAGING - Prescribed packaging is a non-DOT specification pressure vessel designed and manufactured in accordance with the DOT Specification 39 (§§ 178.35 and 178.65), except as follows:

(1) The maximum service pressure at 70EF may not exceed 8500 PSIG. The rated service pressure may not exceed 80 percent of the test pressure and the water volume of each pressure vessel may not exceed one liter.

Continuation of DOT-SP 11650 (22nd Rev.) Page 3

April 23, 2008

(2) Material of construction must conform to all requirements of ' 178.65(b), except that for inflators up to 5,000 psig service pressure, a maximum ladle check analysis carbon content of 0.20 percent is authorized. The chemical composition and mechanical strength properties must conform to documents on file with OHMSPA.

(3) Manufacturing requirements must conform to all requirements of ' 178.65(c).

(4) The minimum wall thickness must be such that the wall stress meets the requirements of ' 178.65(d).

(5) Openings and attachments must conform to all requirements of ' 178.65(e) except that a sidewall opening as shown on Autoliv ASP, Inc. drawing 850008 dated February 28, 1996, drawing E009267 dated December 5, 2000, drawing E024437 dated May 28, 2002 or drawing E 187383, dated September 21, 2007 on file with the Office of Hazardous Materials Special Permits and Approvals (OHMSPA) is authorized.

(6) Each pressure vessel must be equipped with a pressure relief device designed to meet all the requirements for a rupture disk prescribed in the Compressed Gas Association (CGA) Pamphlet S-1.1. The pressure relief device must be capable of preventing rupture of the pressure vessel when subjected to fire test conducted in accordance with CGA Pamphlet C-14.

b. TESTING -

(1) Each pressure vessel must be tested as required in ' 178.65(f) except that -

(i) the hold time at test pressure specified in ' 178.65(f)(1) may be limited to that which is adequate to ensure compliance with the

requirements contained in ' 178.65(f)(1), and

(ii) the maximum duration of the shift specified in ' 178.65(f)(3) may be extended beyond 10 hours at the discretion of the independent inspector.

Continuation of DOT-SP 11650 (22nd Rev.) Page 4

April 23, 2008

(iii) for inflators utilizing ACH-4.1 technology, the lot must be rejected if during the pressure test a failure occurs at a gage pressure less than 1.6 times the test pressure. Autoliv must submit the test data on the first 5 lots of inflators utilizing ACH-4.1 technology to OHMSPA prior to the shipment of the lot.

(2) The flattening test specified in ' 178.65(g) is not required.

(3) A representative vehicle safety system, packaged as it would be for shipment, must be activated and no materials other than non-toxic, non-flammable vapors or gases may be expelled from the package.

c. MARKING -

Each pressure vessel must be durably marked as follows:

DOT-SP 11650/85001  
lot No. xxxxx2  
Manufacturer's Name

This Pressure Vessel May Not Be Refilled

1 Where 8500 represents the design service pressure.

2 Where xxxxx is the lot number as appropriate.

Note: Each line of these markings may be placed without regard to location or order on the pressure vessel.

8. SPECIAL PROVISIONS:

a. In accordance with the provisions of Paragraph (b) of ' 173.22a, persons may use the packaging authorized by this special permit for the transportation of the hazardous materials specified in paragraph 6, only in conformance with the terms of this special permit.

b. A person who is not a holder of this special permit, but receives a package covered by this special permit, may reoffer it for transportation provided no modification or change is made to the package or its contents and it is offered for transportation in conformance with this special permit and the HMR.

Continuation of DOT-SP 11650 (22nd Rev.) Page 5

April 23, 2008

- c. This special permit is limited to pressure vessels used as components of a vehicle safety system. The pressure vessels are excepted from the requirements of the HMR, Part 178 when the design has been certified by an Independent Inspection Agency, approved under ' 107.803 as having met all the requirements of this special permit.
- d. The Independent Inspection Agency's design certification must include test results and documents related to explosive classification and approval. A copy of the certification must be maintained at each facility where the vehicle safety system is manufactured and by the Independent Inspection Agency for a period of 15 years from the date of completion of the design certification.
- e. A current copy of this special permit must be maintained at each facility where the package is manufactured under this special permit. It must be made available to a DOT representative upon request.
- f. Autoliv ASP, Inc. must comply with all provisions of this special permit, and all other applicable requirements contained in the HMR, Parts 100-180. No modifications may be made to the pressure vessel, pyrotechnic components or production vehicle safety system which would affect the performance of the vehicle safety system or its compliance with the requirements of this special permit until such modifications have been reviewed, tested and certified by an Independent Inspector as meeting the requirements of this special permit.
- g. Except when transported on passenger carrying aircraft, devices utilizing the non-DOT specification pressure vessel authorized herein are exempt from the requirements of 49 CFR Parts 100-180 when installed in a motor vehicle or in completed vehicle components such as steering columns or door panels.
- h. Pressure vessels, components, and vehicle safety systems must be transported in strong outside packaging in accordance with ' 173.301(a)(9).
- i. Packagings permanently marked "DOT-E 11650", prior to October 1, 2007 may continue to be used under this special permit for the remaining service life of the packaging or until the special permit is no longer valid. Packagings marked on or after October 1, 2007 must be marked "DOT-SP 11650".



Continuation of DOT-SP 11650 (22nd Rev.)  
Page 6

April 23, 2008

9.

MODES OF TRANSPORTATION AUTHORIZED: Motor vehicle, rail freight, cargo vessel, passenger-carrying aircraft (may not exceed the quantity limitation specified in ' 172.101, column 9A), and cargo aircraft only.

This special permit is to serve as an authorization of The Competent Authority for the United States (CA-9702002) in accordance with the General Packing Instructions Part 3, Chapter 2 Paragraph 2.5 of the International Civil Aviation Organization Technical Instructions (ICAO TI) and additionally meets the requirements of State Variation US 6. Pressure vessels or vehicle safety systems complying with this special permit are authorized to be shipped pursuant to Packaging Instruction 917 of ICAO TI.

10.

MODAL REQUIREMENTS: A current copy of this special permit must be carried aboard each cargo vessel, aircraft or motor vehicle used to transport packages covered by this special permit. The shipper shall furnish a copy of this special permit to the air carrier before or at the time the shipment is tendered.

11.

COMPLIANCE: Failure by a person to comply with any of the following may result in suspension or revocation of this special permit and penalties prescribed by the Federal hazardous materials transportation law, 49 U.S.C. 5101 et seq:

o

All terms and conditions prescribed in this special permit and the Hazardous Materials Regulations, 49 CFR Parts 171-180.

o

Persons operating under the terms of this special permit must comply with the security plan requirement in Subpart I of Part 172 of the HMR, when applicable.

o

Registration required by § 107.601 et seq., when applicable.

Each "Hazmat employee", as defined in § 171.8, who performs a function subject to this special permit must receive training on the requirements and conditions of this special permit in addition to the training required by §§ 172.700 through 172.704.

Continuation of DOT-SP 11650 (22nd Rev.)  
Page 7

April 23, 2008

No person may use or apply this special permit, including display of its number, when this special permit has expired or is otherwise no longer in effect.

Under Title VII of the Safe, Accountable, Flexible, Efficient Transportation Equity Act: A Legacy for Users (SAFETEA-LU)- "The Hazardous Materials Safety and Security Reauthorization Act of 2005" (Pub. L. 109-59), 119 Stat. 1144 (August 10, 2005), amended the Federal hazardous materials transportation law by changing the term "exemption" to "special permit" and authorizes a special permit to be granted up to two years for new special permits and up to four years for renewals.

12.

REPORTING REQUIREMENTS: Shipments or operations conducted under this special permit are subject to the Hazardous Materials Incident Reporting requirements specified in 49 CFR ' ' 171.15 B Immediate notice of certain hazardous materials incidents, and 171.16 B Detailed hazardous materials incident reports. In addition, the grantee(s) of this special permit must notify the Associate Administrator for Hazardous Materials Safety, in writing, of any incident involving a package, shipment or operation conducted under terms of this special permit.  
Issued in Washington, D.C.

for Theodore L. Willke  
Associate Administrator for Hazardous Materials Safety

Address all inquiries to: Associate Administrator for Hazardous Materials Safety, Pipeline and Hazardous Materials Safety Administration, Department of Transportation, Washington, D.C. 20590. Attention: PHH-31.

Copies of this special permit may be obtained by accessing the Hazardous Materials Safety Homepage at

[http://hazmat.dot.gov/sp\\_app/special\\_permits/spec\\_perm\\_index.htm](http://hazmat.dot.gov/sp_app/special_permits/spec_perm_index.htm)

Photo reproductions and legible reductions of this special permit are permitted. Any alteration of this special permit is prohibited.

PO: MMToughiry

## 8 DISTRIBUTION

DTRA C. Lehner, J. Arneson, M. Wroble

Electronic Copies

1 MS1374 R. Rhoades	06723
1 MS1374 K. Seager	06723
1 MS1080 T. Parsons	01749
1 MS1374 P. Bennett	06723
1 MS0964 R. Mata	05702
1 MS0886 D. Derzon	01822
1 MS9104 S. Ferko	08136
1 MS01011 P. Rockett	01011
1 MS9292 N. Gleason	08125
1 MS9004 W. Ballard	08100
1 MS9402 J. Goldsmith	08772
1 MS9406 J. Lund	08132
1 MS0959 D. Chinn	02452
1 MS1080 Mark S. Derzon	1749-2
1 MS1080 Paul Galambos	1749-2
1 MS1080 Keith Ortiz	1749-2
1 MS1196 Gordon A. Chandler	1677
1 MS1316 Shawn B. Martin	1415
1 MS9292 Ronald F. Renzi	8125
2 MS9018 Central Technical Files	8944
2 MS0899 Technical Library)	4536

For Patent Caution reports, add:

1 MS0161 Patent and Licensing Office(electronic)	11500
1 MS0161 Kevin Bieg (electronic)	11500

### References

<sup>1</sup> OUO report, Mark S. Derzon, Gordon A. Chandler, Douglas A. Chinn, Dora K. Derzon, Paul C. Galambos, Clint A. Hall, Robert Koudelka, Shawn B. Martin, Jaime L. McClain, Kenneth A. Peterson, Kenneth R. Pohl, Ronald F. Renzi, Timothy S. Turner, John D. Williams, and William P. Ballard, SAND2007-7584, "Thermal Neutron Detection Using Pixelated High Pressure <sup>3</sup>He Sensors".

<sup>2</sup> Chandler, Gordon Andrew; Martin, Shawn Bryan; Renzi, Ronald F., Derzon, Mark S., "Innovative high pressure gas MEM's based neutron detector for ICF and active SNM detection.", SAND2007-7177. <http://www.prod.sandia.gov/cgi-bin/techlib/access-control.pl/2007/077177.pdf>

<sup>3</sup> Glenn Knoll, 'Radiation Detection Measurement', John Wiley & Sons, Inc. 1989, page 485.

