STUDENT SUMMER INTERNSHIP TECHNICAL REPORT

Mechanical Properties Permanent Foaming Fixatives for D&D Activities

DOE-FIU SCIENCE & TECHNOLOGY
WORKFORCE DEVELOPMENT PROGRAM

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ABSTRACT

The objective of this research was to determine the mechanical performance of polyurethane foam candidates best suited to be permanent foaming fixatives. Multiple polyurethane foams were tested: both flexible and rigid foams were benchmarked and were a combination of fire-rated, non-rated, and intumescent foams. The intumescent foam I-R2 proved to be best candidate in terms of compression, tensile, and adhesion properties. Approximately 40 kN (8000 lbf) was required to compress this foam to 13% its thickness, 3.4 MPa in tension to fracture it, and withstood 2500 N for adhesion testing. The I-R2 foam in a 304 stainless steel pipe took approximately 3000 pounds of force to compress completely out of the pipe. All testing was ASTM standardized with accidental impacts in mind to prevent a release of residual contamination in an operational scenario. All findings can potentially update ASTM E3191 *Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination* with an emphasis on Section 5 which outlines the mechanical performance of a permanent foaming fixative.

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1. INTRODUCTION

Operational Requirement

There is a high operational requirement across the DOE-EM complex for a fixative that can immobilize residual contamination and/or encapsulate three-dimensional void volumes (pipes, glove boxes, drums, etc.) during deactivation and decommissioning (D&D) activities. DOE sites such as Hanford [1] and Idaho National Laboratory [2] can benefit from this technology since both sites contain hundreds of miles of contaminated piping. Savannah River Site (SRS) can also implement this technology since it contains multiple contaminated gloveboxes [3] and hot cells [4] with equipment that cannot be readily removed. Application of this fixative must be simple, cost-effective, and safe in order to ensure worker's safety.

Basis of Interim Operation (BIO) documents postulate contingency scenarios involving seismic activity that can occur at DOE sits during D&D (Figure 1). These events insinuate that current and future fixative technologies must have sufficient mechanical properties to withstand earthquakes, blunt trauma, free fall, etc. in order to prevent a potential release of residual contamination [5].

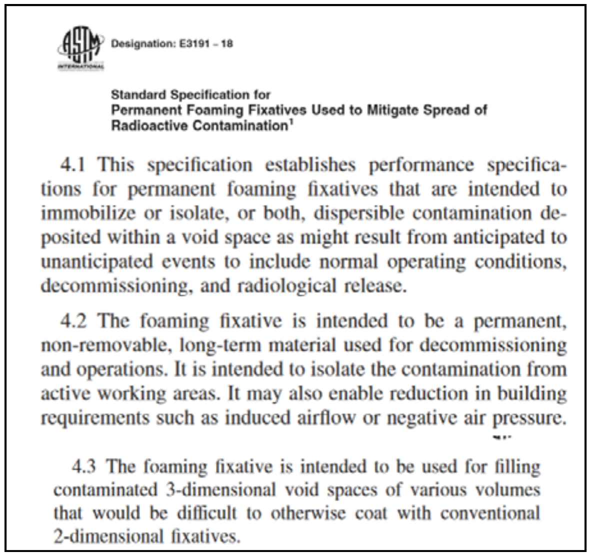
Table 1. Types of Accidents (and Frequencies) Summarized

DOE Site/Facility	Fire Events	Explosion Events	Loss of Confinement (Spill) Events	Natural Phenomena Hazards	Other Events
RFETS Bldg 440	• ***1,200 Drum Fire (EU)*** • ***15 Crate Fire (U)*** • Truck Fire (EU)		• LLW Repack Spill (U) • Drum Spill (A)	• ***Earthquake Collapse (U)***	• ***Aircraft Crash (EU)***
RFETS Bldg 664	• ***3 Drum Fire (U)*** • ***15 Crate Fire (U)*** • *336 Drums + 72 Crates Fire (EU)* • Truck Fire (EU)		• *Multi-Container Drop*	• ***Earthquake Collapse (U)***	• ***Aircraft Crash (worst-case) (EU)*** • ***Aircraft Crash (realistic case) (EU)***
SRS APSF	• *Accountability Msmt. Room Fire (U)*	• *Explosion in Repackaging Area (A)*		• *Seismic Induced Full Facility Fire (U)*	
SRS HB-Line	• Full Facility Fire (EU) • Full Facility Fire & Secondary Events (EU) • ***Intermediate Fire (U)*** • ***Intermediate Facility Fire & Secondary Events (EU)***		• Spill (A)	• Earthquake with Secondary Events (EU)	
SRS Bldg 235-F	• Fire – Best Case (U) • Fire – Worst Case (U)			• Design Basis Earthquake (EU)	
SRS SWMF	• ***TRU Pads - Internal Culvert Drum Fire (U)***	• ***TRU Pads - Culvert Explosion (U)***	• TRU Pads - High Energy Vehicle Impact (EU) • ***TRU Pads - Dropped Steel Box (A)***	• TRU Pads -Tornado (EU)	• 634-7E Buried Waste Helicopter Crash (EU)
Hanford WRAP Facility	• *4 Drum Fire (U)* • *Single Drum Fire in Glovebox (U)*	• ***Drum Explosion with 4 Drum Fire (U)*** • *Single Drum Explosion in Glovebox (U)*	• Solid Waste Box Failure (A)	• ***Design Basis Earthquake (U)*** • Beyond DBE (EU)	
INEEL RWMC	• ***Vehicle Fire (U)***	• *Drum Explosion (A)*	• *Box Spill (A)*	• Design Basis Earthquake (U)	
LANL RAMROD Facility	• Small Fire (A) • Medium Fire (EU) • Large Fire (EU)	• Small Natural Gas Explosion (A) • Large Natural Gas Explosion (EU)	• Coring Glovebox Spill (A)	• Design Basis Earthquake (U)	• Aircraft Crash (EU)

Note: Scenarios in *Italics* are risk dominant events, based on Risk Class I or II for the collocated worker. ***Bold Italics*** denotes that it is also risk dominant for the public.

Figure 1: Basis of Interim Operation Document

Discussions with representatives from the Savannah River National Laboratory (SRNL) have led to a possible solution that satisfies these operational requirements by implementing commercial-off-the-shelf (COTS) polyurethane (PU) foams as permanent foaming fixatives. ASTM E3191 *Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination* was recently developed for the intended use of this technology. Figure 2 describes what a permanent foaming fixative is per ASTM E3191 [6].


Designation: E3191 – 18

Standard Specification for
Permanent Foaming Fixatives Used to Mitigate Spread of
Radioactive Contamination[1]

4.1 This specification establishes performance specifications for permanent foaming fixatives that are intended to immobilize or isolate, or both, dispersible contamination deposited within a void space as might result from anticipated to unanticipated events to include normal operating conditions, decommissioning, and radiological release.

4.2 The foaming fixative is intended to be a permanent, non-removable, long-term material used for decommissioning and operations. It is intended to isolate the contamination from active working areas. It may also enable reduction in building requirements such as induced airflow or negative air pressure.

4.3 The foaming fixative is intended to be used for filling contaminated 3-dimensional void spaces of various volumes that would be difficult to otherwise coat with conventional 2-dimensional fixatives.

Figure 2. Section 4 of ASTM E3191

This technical report is part of FIU's continuing efforts (as well as supporting Mr. Simoes-Ponce's Master's of Science in Mechanical Engineering Thesis) while working closely with Savannah River Site (SRS) and SRNL to address the mechanical performance of permanent foaming fixatives while exposed to physical stressors. This test plan will utilize ASTM E3191: *Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination* as a guide to conduct mechanical property testing. Components of the experimental designs presented in this test plan will be submitted to ASTM E10.03 for review and consideration for revising ASTM E3191.

Relevancy

The relevancy of immobilizing residual contamination cannot be taken lightly. It is common after gross decontamination efforts for sites to remain dormant for multiple years before the final disposition stages. Whatever fixative technology is chosen for implementation must have excellent long-term adhesion capabilities in order to immobilize residual contamination. Release of residual contamination is an issue that has recently been broadcasted nationwide by media outlets. At the end of last year, radioactive dust was found in communities around Hanford Nuclear Site, Rocky Flats and Los Alamos National Laboratory [7]. Microscopic particles of plutonium, thorium, and uranium were found in household dust, automotive air cleaners, and hiking trails. These particles present hazards that can exceed lifelong doses and can make areas around sites uninhabitable. Last year, cleanup missions at Hanford were stopped after plutonium dust was released from a building during demolition activities, became airborne, and coated nearby workers' cars. The fear is now that some of the material was deposited in the Columbia River, which can present a nationwide catastrophe [8].

Past Research of Polyurethane Foams

Polyurethane foams have been examined by SRNL and FIU-ARC for possible application to support D&D activities for some time now. The properties of polyurethane foams can be enhanced by altering the fabrication process. Polyurethane foam products are also known for their ability to encapsulate a variety of small spaces which could be conducive in radioactive environments, such as within piping or a glove box. Polyurethane foams are already being considered as fixatives but have yet to be implemented in an operational environment [9].

SRNL Radiological Shielding Tests

Scientists at SRNL have recently been interested in conducting research and experiments on rigid and flexible polyurethane foams to determine their ability to immobilize radioactive contamination. Rigid foams are known for reducing energy costs and for being great insulation materials while flexible foams are used in a wide variety of products like bedding and shock absorbers due to their flexibility. SRNL initiated testing on a wide variety of COTS foams to determine which properties would be deemed compatible with the safety basis requirements in a radioactive environment. The properties deemed relevant were the foaming characteristics, temperature profile, and loading of high-density additives to shield gamma isotopes such as Am-241, Cs-137, and Co-60.

SRNL created and cured the following four foam samples with and without additives: Foam - It 3, Foam – It 8, Flex Foam – It III, and Flex Foam It- 25. All the foam samples were within about ±5% of their expected expansion volume. Foams with the following additives experienced a slight temperature difference compared to the unmodified foams: bismuth, bismuth oxide, tungsten oxide, sodium tungstate, barium chloride, and barium sulfate. The temperature difference was within the margin of error and all of the foams cooled to room temperature within 15 minutes. Using an identiFINDER, an experiment using the various additives was conducted by SRNL to test the foams' abilities to shield radiation. The results demonstrated the following effective shielding of the gamma isotopes: 98% of Am-241, 16% of Cs-137, and 9.5% of Co-60. Among the tested additives, bismuth and bismuth oxide provided the best shielding [10].

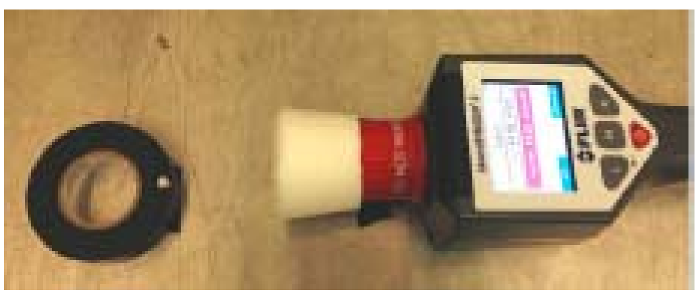


Figure 3. SRNL radiation shielding testing with a 10 cm distance between the source and identiFINDER.

FIU-ARC Fire Testing

In parallel research efforts, FIU-ARC conducted flammability tests during Performance Year 8 under the DOE-EM Cooperative Agreement to address the thermal resiliency of 6 COTS polyurethane foams (i.e., no additives). Two of the tested foams were fire-rated by their manufacturers, two were non-fire rated, and the final two were intumescent products. The flame tests were loosely based on a near-fit standard (IEC 60695-111-10) in which the foam sample would be subjected to a two-hour flame produced by a butane torch. All samples disintegrated within minutes except for the I-F4 and I-R2 intumescent foams, which are able to produce an insulating soft char that is 50-100 times the foam's original volume. The I-R2 sample was determined to be best-in-class due to its ability to maintain its structural integrity, demonstrate enhanced thermal insulation, produce limited to no smoke, and mitigate flame spread and propagation. This product was able to withstand hours of direct flame without significant degradation [11].

Figure 4. FIU-ARC flame testing on foam – F-2.

Mechanical Testing

Polyurethane foams have been used as an impact limiter in nuclear packaging for over 30 years. SRNL have been conducting a variety of stress and mechanical testing on polyurethane foams used in current Model 9977 shipping packaging to limit impact and protect any hazardous material inside [12]. The 9977's purpose is to ship plutonium and uranium in metal and oxide form. Mechanical testing is done on these containers and polyurethane foams to ensure no release occurs during accidental impact that are associated with regulatory Normal Conditions of Transport and Hypothetical Accident Conditions. Testing involves vibration, water spraying, free drop, crush, and puncture testing. All mechanical testing complies with Section III of the American Society of Mechanical Engineers (ASME). Figure 5 shows two types of containment vessels (CV) that correlates with Model 9977 shipping containments and both containment vessels contain large amounts of polyurethane foams.

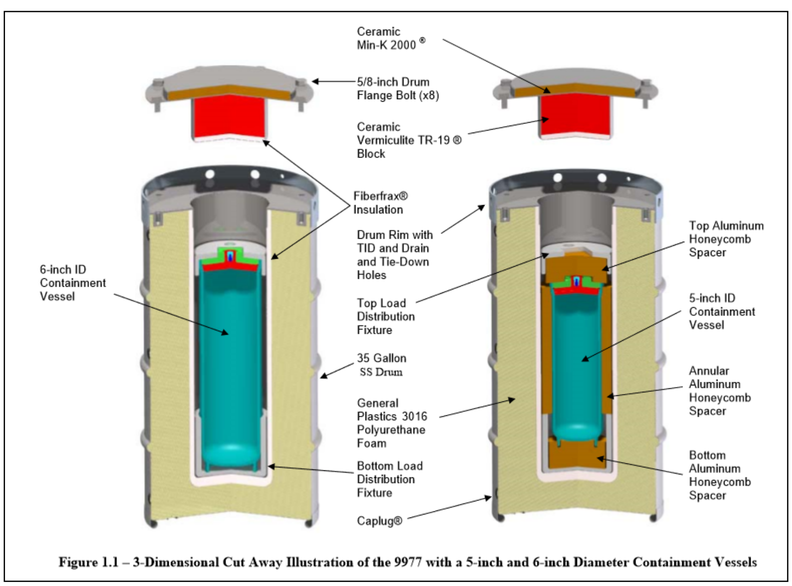


Figure 5: 5CV AND 6CV of Model 9977

Sandia National Laboratory (SNL) have performed mechanical testing on polyurethane foams within the past couple of years. SNL conducted compression testing on different density rigid foams and learned that the denser rigid foams produced higher yield points and stresses. SNL also confirmed that temperatures above 75 degrees °F decreased the foams' compressive strengths (300-400 degrees °F). SNL also concluded the tensile strengths were about 80% of the compressive strengths. Creep testing by SNL determined how these rigid foams would hold up long term and proved these foams should not exceed 40% of the strength at 10% strain [13].

SNL also performed mechanical testing on flexible foams as well. They reported that once these flexible foams are cooled to temperatures below their glass transition temperatures (-35 °C), they act like rigid polyurethane foams and can now plastically deform. Different temperatures above and below the glass transition temperatures also drastically alter the mechanical performance of flexible foams. SNL ultimately concluded that flexible foams would reach their original shape if operated at room temperature. SNL created a foam model to help predict scenarios for accidental impacts and also found that if a foam were to experience a large loading, its performance would likely weaken afterwards [14].

An adhesion study on polyurethane foams on thermoplastic materials was conducted in Germany in 2005 [15]. The study states different methods like plasma treatment and priming surfaces can improve adhesion capabilities but in an operational sense, it is best to not do any pretreatment for workers' conveniences. A peel test was conducted on the polyurethane foams and Figure 6 displays the three different failure modes that can occur.

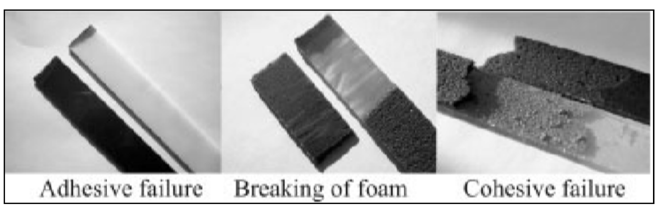


Figure 6: Left: Foam Completely delaminates off substrate; Middle: Foam still adheres onto substrate; Right: Both foam and substrate experience failure

Breaking of foam and cohesive failure are preferred in an operational sense since the foam still adheres onto the substrate and will still somewhat immobilizes residual. Figure 7 shows a graph of what occurs during the stages of the peel-off test.

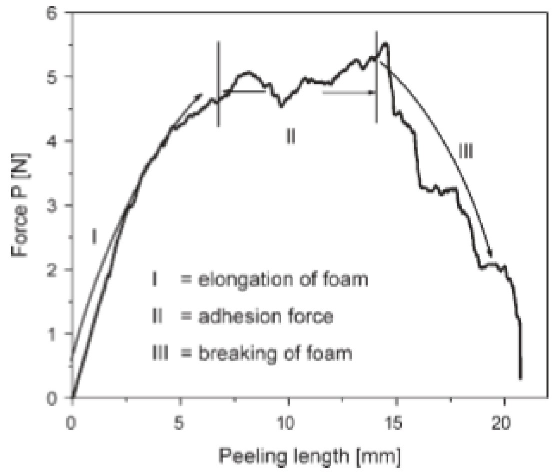


Figure 7: Peel Off Test Results

Initial mechanical testing by SRNL and FIU-ARC, in alignment with ASTM E3191, helped baseline a variety of polyurethane foams that are non-fire rated, fire rated, and intumescent. Initial findings from last summer at SRNL determined that intumescent polyurethane foams (Figure 8) were the best in class in terms of tensile strength, glass transition temperature, strain at failure, and decomposition temperatures shown in Table 1 and Figure 9 [16]. The intumescent foams, I-R2 and I-F4, were initially identified as possible candidates as a permanent foaming fixative due to their mechanical and fire retardancy capabilities.

Figure 8: Intumescent foams to be used in this experiment (I-R2 and I-F4)

Table 1: Tensile Testing Data

Foam Sample	Young's Modulus (N/mm^2)	Peak Stress (N/mm^2)	Peak Load (N)	Strain at Break (mm/mm)
F1	0.202	0.183	7.39	0.93
F2	0.132	0.120	5.375	1.137
F3	0.180	0.233	11.57	1.557
I-F4	0.272	0.027	2.336	0.149
R1	**50.40**	**1.30**	52.05	**0.03**
I-R2	42.7	0.88	**74.89**	**0.03**

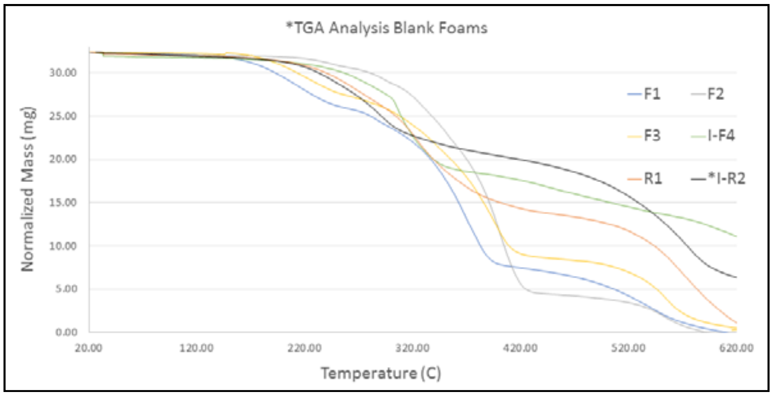


Figure 9: TGA Graph

2. EXECUTIVE SUMMARY

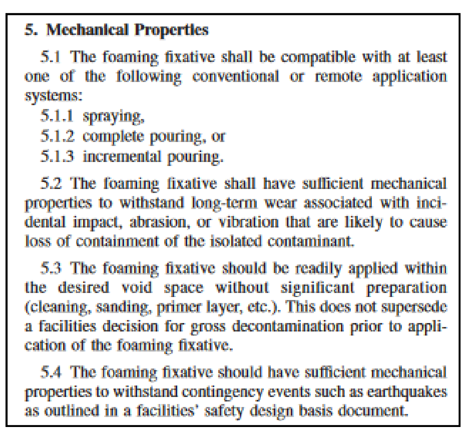
This research work has been supported by the DOE-FIU Science & Technology Workforce Initiative, an innovative program developed by the US Department of Energy's Environmental Management (DOE-EM) and Florida International University's Applied Research Center (FIU-ARC). During the summer of 2019, a DOE Fellow intern Tristan Simoes-Ponce spent 11 weeks doing a summer internship at the Savannah River Site under the supervision and guidance of Dr. James Connor Nicholson, PhD. The intern's project was initiated on May 18, 2019 and continued through August 3, 2019 with the objective of characterizing the mechanical properties of polyurethane foams.

3. RESEARCH DESCRIPTION

This test plan primarily addresses concerns of mechanical degradation that can cause the permanent foaming fixative to delaminate off a substrate and induce the release of residual contamination in enclosed operational volumes. The first test objective is to identify the mechanical properties of 6 COTS polyurethane foams in terms of tensile and compressive strength. The second objective is to test the adhesive strength of the foams to a 304 stainless steel substrate in tension. The last objective is to test the adhesion of the best permanent foaming fixative candidate in an operational volume while subjected to compression. The COTS foam products (Figure 10) that will be used include two intumescent PU foams (I-F4 and I-R1) and four non-intumescent PU foams (F1, F2, F3 and R1). The I denote intumescence, F denotes flexible and R denotes rigid in this naming convention.

Figure 10: Foams to be Tested On (F1, R1, F2, F3, I-F2, R2)

All findings from this demonstration will be published as a technical report and will be used in support of Tristan Simoes-Ponce's Master's of Science in Mechanical Engineering Thesis. It is intended for this research to lead to more substantive advancements in the concept of permanent foaming fixatives to improve safety in D&D activities across the entire DOE EM complex. The outcome of this test plan can potentially update the performance criteria of Section 5 of ASTM E3191: *Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination* (Figure 11).



5. Mechanical Properties

5.1 The foaming fixative shall be compatible with at least one of the following conventional or remote application systems:

5.1.1 spraying,

5.1.2 complete pouring, or

5.1.3 incremental pouring.

5.2 The foaming fixative shall have sufficient mechanical properties to withstand long-term wear associated with incidental impact, abrasion, or vibration that are likely to cause loss of containment of the isolated contaminant.

5.3 The foaming fixative should be readily applied within the desired void space without significant preparation (cleaning, sanding, primer layer, etc.). This does not supersede a facilities decision for gross decontamination prior to application of the foaming fixative.

5.4 The foaming fixative should have sufficient mechanical properties to withstand contingency events such as earthquakes as outlined in a facilities' safety design basis document.

Figure 11: Section 5 of ASTM E3191

Fabrication of Polyurethane Foam Samples

Tensile Testing Samples

Table 2 shows how much of each component were used to develop the tensile testing samples. The intumescent foams were not included because there is no fixed amount to apply them due to them hardening and dispersing so fast. Overcompensation of amounts of components were used to ensure quality performance.

Table 2: Tensile Testing Mixing Components Ratios

Tensile Testing				
Foam Identifier	**Volume (mL)**	**A (mL)**	**B (mL)**	**Ratio (A:B)**
F1	50	10	10	1
R1	86	16	8	2
F2	50	12	24	0.5
F3	50	11.5	13.5	0.85

A mold was 3D printed for the rigid polyurethane foams with dimensions that complied with the Type B tensile dye of ASTM D1623 *Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics* [17]. The mold was taped around all its edges and was placed on wax paper (Figure 12). Using the I-R2 foam dispenser gun, foam was poured inside the mold at a quick rate. Once the foam started to expand, a 12-inch by 12-inch 304 stainless steel coupon was placed on top of the mold sandwiching the foam inside the mold. After a couple of minutes passed, the large 304 stainless steel coupon was taken off and a saw was used to trim any excess foam from the top surface (Figure 12). The foam was then extracted from the mold to produce the tensile testing sample.

Figure 12: I-R2 Fabrication Process

A total of five samples were produced (Figure 13). Two of the samples created will be used as test dummy samples due to them having imperfections and impurities. The other rigid foam, R1, was made in the same manner.

Figure 13: Five tensile testing samples of I-R2

The same process was done for the flexible foams using the amounts of both Part A and B prescribed in Table 1. The 3D mold used is shown in Figure 14 and complies with ASTM 3574 *Flexible Cellular Materials – Slab, Bonded, and Molded Urethane Foams* Test E dimensions [18]. Figure 14 also shows the process of curing and extraction from the mold.

Figure 14: F3 Fabrication Process

A total of five samples were produced for F3 (Figure 15). All the other flexible foams in similar fashion.

Figure 15: F3 Tensile Testing Samples

<u>Adhesion Testing (Tension) Samples</u>
Table 3 shows how much of each component were used to develop the adhesion tensile testing samples. The intumescent foams were not included because there is no fixed amount to apply due to them hardening so fast. Overcompensation of amounts of components were used to ensure quality performance.

Table 3: Adhesion Testing Mixing Amounts of Parts

Adhesion (Tension)				
Foam Identifier	**Volume (mL)**	**A (mL)**	**B (mL)**	**Ratio (A:B)**
F1	131	15	15	1
R1	131	20	10	2
F2	131	16	32	0.5
F3	131	35	41	0.85

A mold was 3D printed with dimensions that complied with Type C specimens in ASTM D1623. The inner dimensions of the rectangular mold were 2.25 inch by 2 inch by 2 inch. The two 304 stainless steel coupons would be placed on the inner walls of the mold while the foam would be dispersed and cured between them (Figure 16).

Figure 16: 3D mold with stainless steel coupons inside

The same process for the tensile testing die was applied as the mold was placed on top of wax paper and a larger 304 stainless steel coupon was used to prevent the foam from pouring out of the mold for the intumescent foams. The other non-intumescent foams used the required amounts as highlighted in Table 3. Once the foams cured and hardened inside the mold, it was extracted from the mold (Figure 17).

Figure 17: R1 Fabrication Process

A total of three samples were produced of R1 (Figure 18). The other five foams were made following the same process.

Figure 18: Four Adhesion Testing Samples of R1

<u>Compression Samples</u>

Table 4 shows how much of each component were used to develop the compression samples. The intumescent foams were not included because there is no fixed amount to apply them due to hardening so fast. Overcompensation of amounts of components were used to ensure quality performance.

Table 4: Amounts to Produced Required Sample

	Compression (Cylinder)				Compression (Square)			
Foam Identifier	Volume (mL)	A (mL)	B (mL)	Ratio (A:B)	Volume (mL)	A (mL)	B (mL)	Ratio (A:B)
F1	102	7	7	1	65	5	5	1
R1	102	10	5	2	65	6	3	2
F2	102	12	24	0.5	65	9	18	0.5
F3	102	23.5	27.5	0.85	65	15	17.5	0.86

Two molds were purchased locally and the dimension of each cavity in each mold complied with standards ASTM 3574-C (flexible) and ASTM D1621 *Compressive Properties of Rigid Cellular Plastics* (rigid) [19]. Both rectangular and circular specimens will be considered.

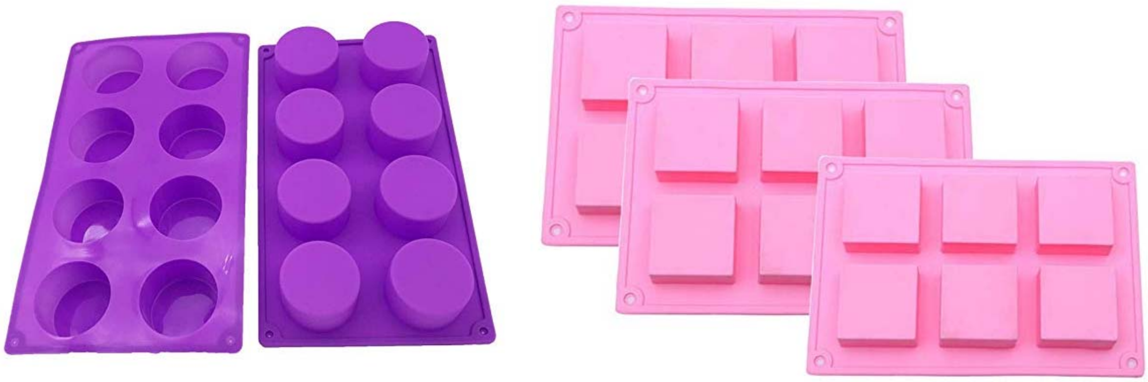


Figure 19: Compression Sample Molds

The same process was used for the intumescent foams, and any excess foam was shaved off. The other foams used the amounts in Table 4 and were stirred and allowed to cure overnight. Figure 20 shows R1 foam samples.

Figure 20: R1 Compression Samples

Compression testing was also performed on the cubic samples after they are used for adhesion testing. Figure 21 showcases the rigid foam samples (R1 and I-R2).

Figure 21: Cubic Adhesion Samples

Experimental Design

<u>Experiment 1A. Tensile Tester</u>

Residual contamination can be trapped inside permanent foaming fixative and tension can cause the release of the residual contamination due to induced delamination. Tensile testing was performed to characterize the foam's mechanical properties that involve Young's Modulus, strain, peak stress and peak load. These properties can be calculated by analyzing the produced stress-strain curves. An MTS Criterion 43 Tensile Tester was utilized for this task. The standards to be considered are ASTM D3574-E *Flexible Cellular Materials – Slab, Bonded, and Molded Urethane Foams* (flexible) and ASTM D1623 *Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics* (rigid). Figure 23 shows the tensile dyes' dimensions for both standards. ASTM D3574-E specifies to use a pull rate of 500 mm per second while ASTM D1623 states to use a pull-rate of 0.05 in per min.

Figure 22: Tensile Testing – ASTM D3574 Test E

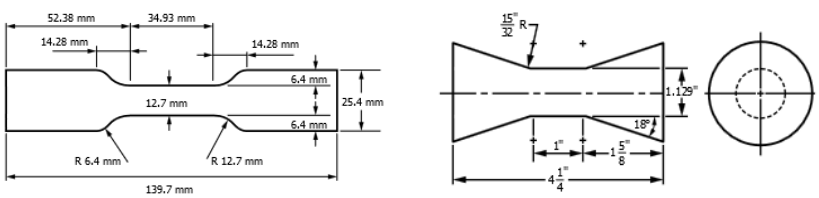


Figure 23: Left picture: Tensile Dye Using ASTM D374-F (Flexible); Right picture: Tensile Dye Using ASTM D1623 (Rigid)

<u>Experiment 1B: Adhesion Testing (Tension)</u>

Adhesion capabilities will ultimately decide whether a permanent foaming fixative can immobilize residual contamination. Any sort of incidental impact can cause the foam to delaminate from the substrate causing residual contamination to be released. ASTM D1623 *Test Method for Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics* will be used to conduct adhesion testing.

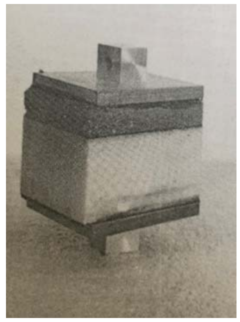


Figure 24: Adhesion Testing Sample per ASTM D1623

Using the MTS Criterion series 43 Tensile Tester, the tensile adhesion strength will be calculated to see the amount of force or stress it requires to pull the foam off the 304 Stainless Steel substrate in 3 to 6 minutes. The standard suggests using a rate of pull of .05 in/min for each inch of test section gauge length. The 304 stainless steel coupons will be 2 inch by 2 inch by .125 inch thickness so the I-R2 foam's dimensions will be 2 inch by 2 inch by 2 inch, making the rate of pull 1 in/min per the standard.

For the tensile testing machine to grip the adhesion sample, some sort of attachment must be applied for the machine to grip. Before attaching any sort of grippers, the outer surfaces of the coupons were sanded thoroughly with 120 grit sand paper to increase the surface energy. Four total hinges were sanded as well and then were super glued using Loctite Super Glue Gel on the top and bottom surfaces as symmetrical as possible to ensure even stress distribution. The hinges were glued on the same axis to prevent any shearing and were left over night to cure (Figure 25).

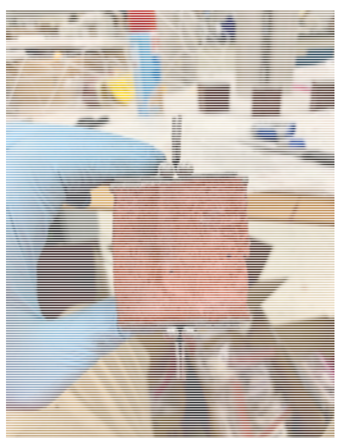


Figure 25: Adhesion Sample with Four Hinges

Preliminary testing was done to ensure the super glue would be strong enough to withstand the pulling. A pull rate of 0.1 inches per minute was used to pull the top 304 stainless steel coupon off the I-R2 foam (Figure 26).

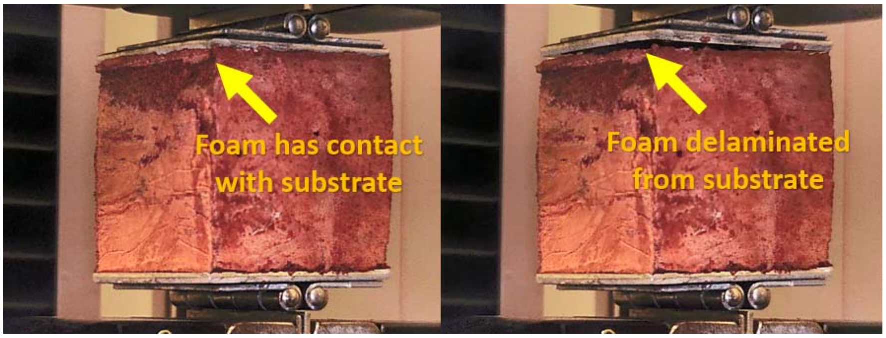


Figure 26: Left Picture: Foam still has contact with substrate before adhesion testing; Right Picture: Foam delaminated from substrate after adhesion testing

Experiment 2A: Compression Testing

Residual contamination may be trapped inside permanent foaming fixative and any sort of static loading can cause the release of the residual contamination. Compression testing will be performed to characterize the foam's mechanical properties that involve compression stress, compression strain, compression modulus and maximum compression modulus. An MTS Criterion 43 Tensile Tester was utilized for this task. The standards that were utilized are ASTM 3574-C *Flexible Cellular Materials – Slab, Bonded, and Molded Urethane Foams* (flexible) and ASTM D1621 *Compressive Properties of Rigid Cellular Plastics* (rigid). ASTM 3574-C for the flexible foams states to pre-flex the foams twice at a rate of 250 mm per minute and then compress the foam for 50 mm per minute and to dwell for a minute. ASTM D1621 states to compress the rigid foam at a rate of 10% of its initial thickness until the foam is compressed 13% of its original thickness. Testing will stop if the rigid foam reaches this strain criteria or until it reaches the load limit of the MTS which was set to 40 kN (8992.35 lbf).

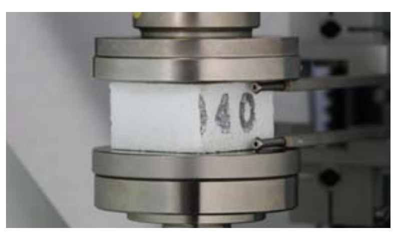


Figure 27: Compression Testing - ISO 844

Experiment 2B: Adhesion Testing (Compression)

The objective was to determine how much compressional force will be required to compress the permanent foaming fixative off an operational volume, which is a 304-stainless steel pipe. If the permanent foaming fixative leaves material in the internal walls of the pipe, it will be concluded that the shear strength will be less the adhesive strength. The opposite can be said if the permanent foaming fixative does not leave any material then the shear strength will be greater than the adhesion strength. The pipe samples are four inches in internal diameter and four inches in length. 2 samples were made in total for preliminary testing.

Figure 28 shows a cross-sectional view of how the permanent foaming fixative was compressed out of the 304 stainless steel pipe and the dimensions of all components involved. An MTS 43 Criterion tensile tester was used with compression plates.

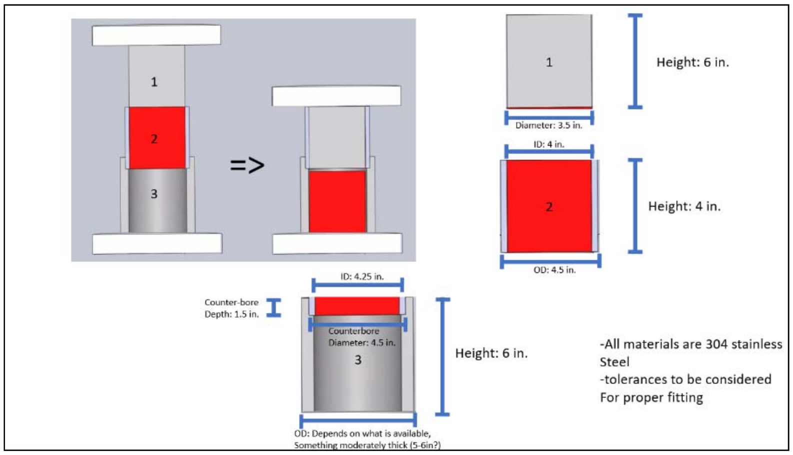


Figure 28: Left picture shows the permanent foaming fixative before compression; Right picture shows the permanent foaming fixative after compression

4. RESULTS AND ANALYSIS

Experiment 1A: Tensile Testing

Figure 29 shows the process of tensile testing for a flexible foam. Flexible foams would typically elongate until sudden rupture occurs. No sign of permanent deformation occurs. Rigid foams display the opposite behavior as they do not stretch and will exhibit permanent deformation. The flexible foams had an average gauge length of 34.93 mm and cross-sectional area of 96.774 mm^2. The rigid foams had to be shaved down because they were too thick for the grips of the machine, and their average gauge length was 25.4 mm and cross-sectional area of 231 mm^2.

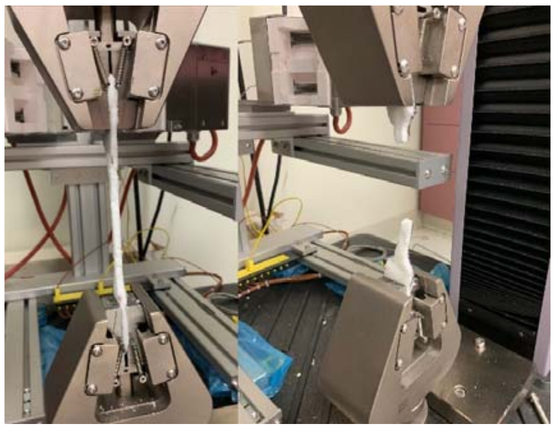


Figure 29: Flexible Foam Tensile Testing Process

Figure 30 displays the produced stress-strain curves. It shows the flexible foams stretched to approximately triple their length. The rigid foams barely stretched and experienced the highest stresses and loadings.

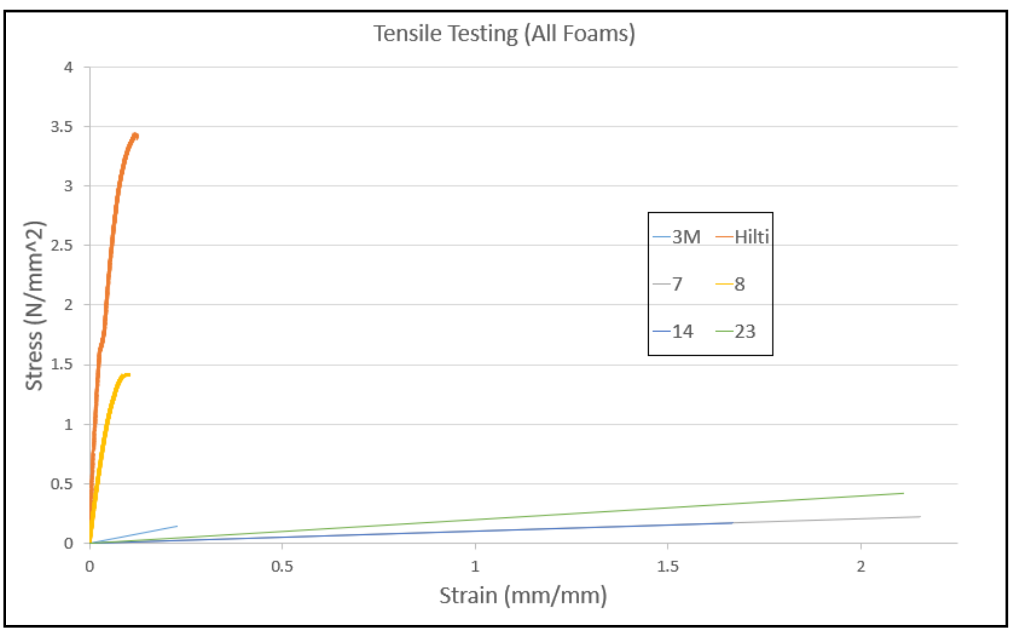


Figure 30: Stress-Strain Graph for All Foams

Both ASTM E3574 and D1623 state to calculate the stresses and loadings that occur at failure as well as the strain. The rigid foams, R-1 and IR-2, had the highest stresses of 1.42 N/mm^2 and 3.44 N/mm^2. The rigid foams also had the highest modulus. The loadings that coincide with these stresses are 328.02 and 794.64 N respectively. The flexible foams had the highest strain values with F-1 extending to 215% its original gauge length. Table 5 displays all findings from this test.

Table 5: Tensile Testing Values

Foam Identifier	Peak Load (N)	Peak Stress (N/mm^2)	Strain at Break (mm/mm)	Modulus (N/mm^2)
F-1	21.52	0.22	2.15	0.10
F-2	16.44	0.17	1.67	0.10
F-3	40.61	0.42	2.11	0.69
IF-4	13.83	0.14	0.23	0.63
R-1	328.020	1.42	0.1	25.5
IR-2	794.640	3.44	0.12	71.85

Experiment 1B: Adhesion Testing (Tension)

Figure 31 illustrates the process of adhesion testing for a flexible foam. Flexible foams also stretched the most compared to rigid foams before the stainless-steel coupon delaminated.

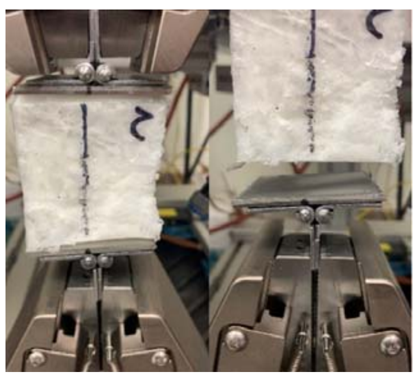


Figure 31: Flexible Foam Adhesion Testing Process

Figure 32 illustrates the results of adhesion testing. The noisy data for the flexible foams is due to using a load cell that can go up to 50 kN. The rigid foams experienced higher stresses (4.657 N/mm^2) before failure while the flexible foams experienced higher strains (22.8%) before failure.

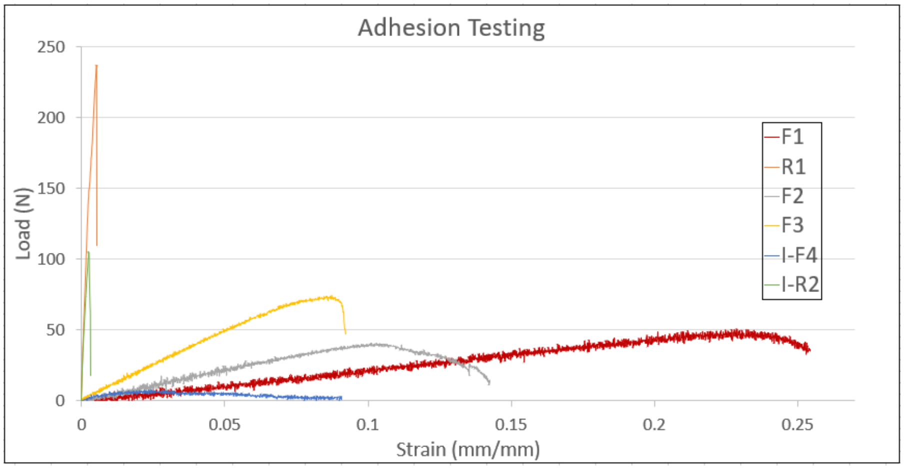


Figure 32: Adhesion Testing Results

Table 6 shows the results from this testing which confirms that the rigid polyurethane foams would be more suited to be used as permanent foaming fixatives. This is due to them reaching higher stresses before delamination occurs which can cause the release of residual contamination in an operational scenario.

Table 6: Adhesion Testing Results

Foam Identifier	Peak Stress (N/mm^2)	Peak Load (N)	Strain % (mm/mm)	Deformed Length (mm)
F-1	0.98	50.22	22.88	62.42
F-2	0.79	40.27	10.05	55.90
F-3	1.46	74.16	8.74	55.24
I-F4	0.13	6.82	2.36	51.99
R1	4.65	236.57	0.51	51.05
I-R2	2.06	104.85	0.25	50.92

From Table 6, R-1 and I-R2 are clearly the best in class in terms of adhesion tensile performance. I-R2, however, did perform better when tested at FIU and reached a max loading value of almost 450 N (101.16 lbf), which is higher than the 236.57 N value obtained at SRNL (left part of Figure 33). One test run at SRNL produced loadings of almost 2500 N (562 lbf) before slipping occurred (right part of Figure 33). These findings show promising results for the intumescent foam (I-R2) in an ideal scenario and will be further investigated.

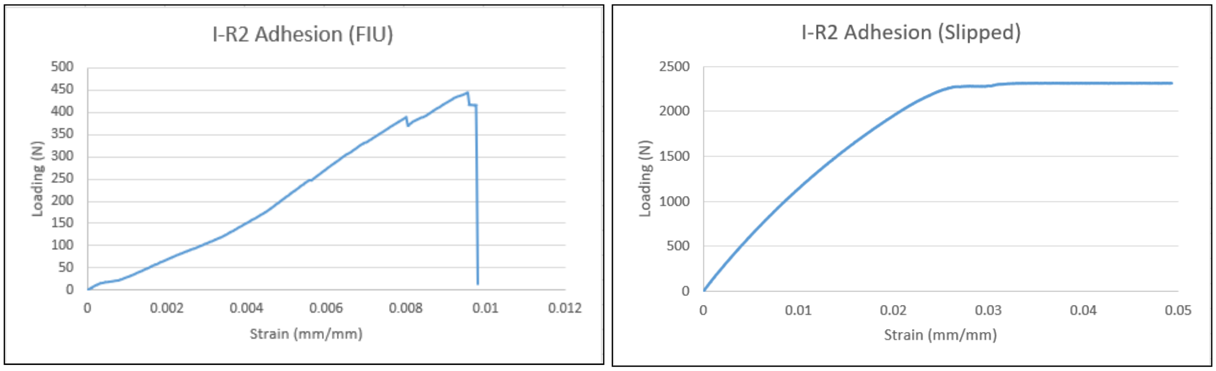


Figure 33: Left: FIU Adhesion Test Run; Right: Slipped Run

Analysis of residue on adhesion test coupons:

Figure 34: Scanned surfaces of stainless steel coupons after adhesion testing

ImageJ was used on the scanned surfaces of the stainless-steel coupons that were pulled off (Figure 34). Analysis of the surfaces helped show which foam still adhered onto the coupon. For samples that had significant residue, this indicates the failure was shearing of the material rather than delamination as the failure mode. Table 7 shows the ImageJ results and illustrates that the intumescent foam, I-F4, adhered the most to the surface (61.45%). This is another advantage intumescent technology has over non-intumescent technology.

Table 7: ImageJ Area Fraction Results

Foam Identifier	Average (%)	Average (in^2)
F-1	4.05	0.16
F-2	0.00	0.00
F3	0.26	0.01
I-F4	61.45	2.46
R-1	7.38	0.30
I-R2	2.84	0.11

Experiment 2A: Compression

Figure 35 illustrates the compression testing for a flexible foam cube.

Figure 35: Flexible Foam Adhesion Testing Process

Figure 36 shows the compression results for the cubic samples. The cubic samples were previously used for the adhesion testing, but all suffered no structural degradation. All the cubic samples were 50.8 mm thick and had a cross-sectional area of 2580 mm^2 which satisfies both ASTM 3574-C and ASTM D1621 dimension requirements. The minimum thickness and cross-sectional areas for ASTM 3574-C are 20 mm and 2500 mm^2, respectively, while the minimum thickness and cross-sectional area for ASTM D1621 is 25.4 mm and 2580 mm^2. Some of the dimensions for cylindrical and rectangular varied slightly and were calibrated appropriately so all the samples had the same dimensions. For the rigid foams, I-R2 and R1, they first experience an initial elastic regime followed by a plateau regime where the loading is nearly constant. In the initial linear elastic part, the rigid foams are compressed uniformly. For the plateau part, they start to plastically deform as the cell walls are compressed together [14].

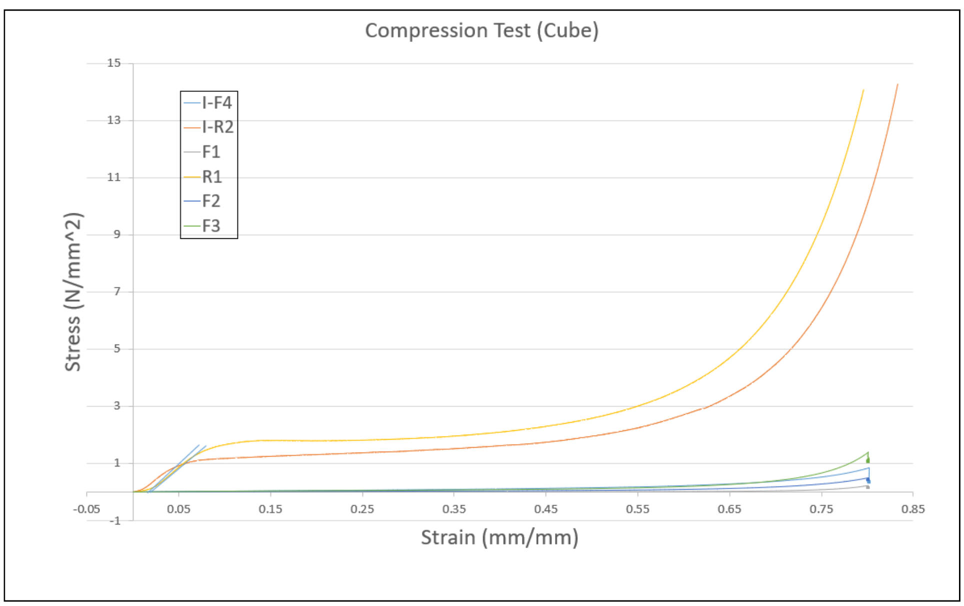


Figure 36: Compression Results (Cube)

Table 8 shows the results for all parameters that ASTM 35474-C requires. It only asked for values at 50% deflection, but 80% deflection values were also obtained. The rigid foams, R-1 and I-R2, performed better as evident in Table 8 and Figure 36 by achieving higher loading and stresses than the flexible foams. The flexible foams got slightly shorter while the rigid foams were able to restore a little bit of thickness once uncompressed.

Table 8: Compression Results for Both Flexible and Rigid Foams (Cube)

Foam Identifier	50% Deflection Stress (MPa)	50% Deflection Load (N)	80% Deflection Stress (MPa)	80% Deflection Load (N)	Thickness Decrease (%)	Final Thickness (mm)
F-1	0.009	23.47	0.22	584.04	3.4	49.09
F-2	0.056	143.70	0.49	1282.56	1.24	50.16
F-3	0.117	301.89	1.38	3583.08	1.29	50.15
IF-4	0.151	389.06	0.83	2144.22	32.41	34.34
R-1	2.579	6652.71	14.079	36325.70	68.42	16.04
IR-2	1.949	5028.88	10.08	26028.43	74.75	12.82

Table 9 highlights the results that ASTM D1621 required. The three stages these rigid foams go through are the linear elastic region, plateau region, and densification. Anything that happens after the initial linear elastic region will result in permanent deformation. The modulus computed, which is the measure of stiffness a material can exhibit before plastic deformation, was greater for the non-intumescent rigid foam than I-R2.

Table 9: Rigid Foam Compression Data (Cube)

Rigid Foam	Zero-Point Reference	10% Stress (MPa)	10% Load (N)	Modulus (MPa)
R1	.017, .09	1.74	4498.36	14.38
I-R2	.0104,.0976	1.19	3068.53	16.84

Compression testing results for the other geometric shapes can be found in the Appendix. The results are similar to the findings for the cubic specimens. The dimensions for the other geometric samples varied, so slight manupilation was applied to ensure they are equal and complied with the standards. Overall, there is no criteria on what compression amount will cause the release of residual contamination so overcompensation was used by assuming 80% deflection. All the rigid foams did not reach the 13% thickness due to the machine limit criteria being reached before (40,000 N). The flexible foams, however, showed degradation while preflexing, which could cause the release of residual contamination (Figure 37). The intumescent flexible foam, I-F4, did not return to its original height after preflexing which can cause additional concerns.

Figure 37: Structural Degradation of Flexible Foams

Experiment 2B: Adhesion Testing (Compression)

Figure 38 shows the experimental design Figure 28 proposed. The machine shop at SRNL machined the components to the dimensions prescribed in Figure 28. A rate of 0.4 inches per minute was used since this is 10% of the pipe's height. This procedure is not standardized, but follows some protocols described in ASTM D1621. The I-R2 was the foam of choice due to it passing rigorous fire testing and displaying promising mechanical performances.

Figure 38: Pipe Adhesion Experimental Design

Figure 39 is the output load vs displacement graph with the two samples. Both curves follow the same pattern Figure 9 displays. The curves shoot up, stay somewhat constant, and then drop. Trial One took a max load of 2831 pounds of force while Trial Two took 240 pounds of force. This disparity might be because Trial One was made with the beginning of the I-R2 foam cartridge and Trial Two was made with the end. More testing of more samples can prove this.

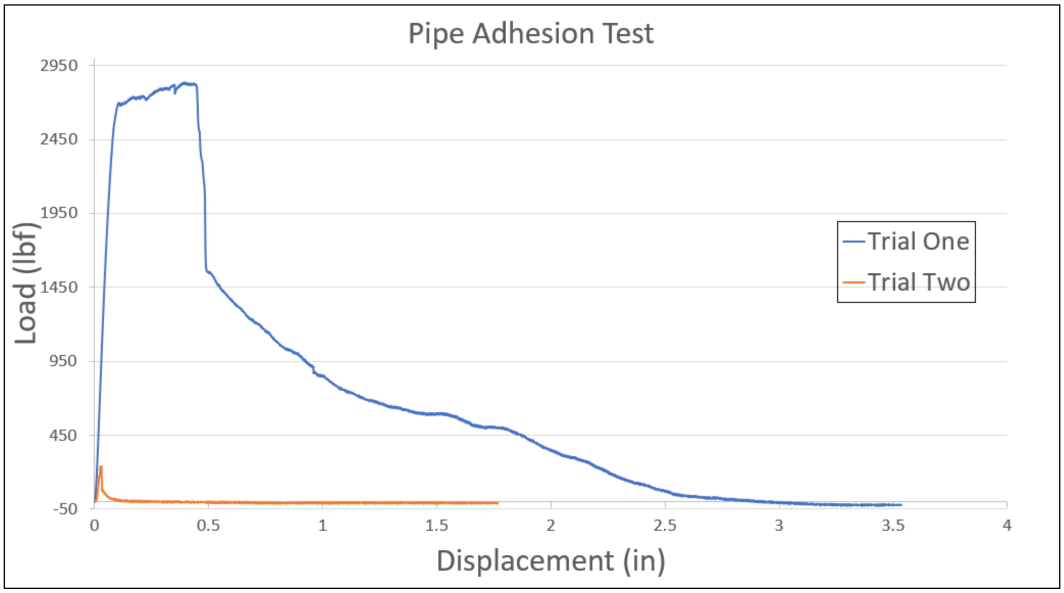


Figure 39: Adhesion (Compression) Graph

Figure 40 shows the samples after testing. Trial One had some material still on the inner walls of the pipe while Trial Two plunged out uniformly. In an operational sense, it is recommended to have some material still adhering onto the substrate, so it can still immobilize residual contamination. It also speaks to the failure mode of the material: if there is material still left on the walls, the failure was interior to the material (fracture) rather than a complete removal of the material (delamination). The shear stress will be less than the adhesion strength in this case. Trial 1 represents when this occurs while Trial 2 shows the opposite. This discrepancy might be a fabrication and application issue which will require more testing to prove.

Figure 40: Left: Trial One and Trial Two Comparison; Middle: Trial One Aftermath; Right: Trial Aftermath

5. CONCLUSION

The I-R2 foam proved to be the best polyurethane foam out of the six candidates in terms of mechanical properties. It demonstrated excellent compression capabilities by reaching the load limit of the load cell (40 kN~9000 lbf) for all geometric configurations. Tensile testing results showed it experienced the least amount of strain while handling the highest payloads and stresses. The other rigid foam, R1, had better adhesion capabilities but some samples of I-R2 performed better before slipping of grips occurred. These results are promising in terms of adhesion. Almost 3000 pounds of force was required to extract the I-R2 foam out of the 304 stainless steel pipe which means an accidental impact must be greater than that amount for something catastrophic to occur. The intumescent capabilities of the I-R2 is also favorable as it can now withstand extreme heat conditions as well as high payloads, preventing the release of residual contamination.

Future testing could help confirm the down-selection of the I-R2 foam as a permanent foaming fixative. Impact testing can be performed to help solidify the down-selection. Determining what minimum contact the I-R2 shall have with a 304 stainless steel substrate for adequate adhesion results can also be performed. Figure 41 shows an experiment conducted at FIU-ARC. The I-R2 foam was applied in a plastic pipe with some "contamination" inside. The I-R2 foam hardened so fast, it did not bleed into the 'contamination.' The I-R2 foam essentially pinned the 'contamination' onto to the wall and immobilized the 'contamination' in a global sense, but not a local sense. Figure 42 shows some pieces cut from the pipe sample. The permanent foaming fixative adhered well to the substrate but did not adhere well onto the substrate with the 'contamination' sandwiched between.

Figure 41: Left picture: Encapsulation pipe experiment conducted at FIU-ARC; Right picture: Simulated contamination (green) inside pipe with foam (red)

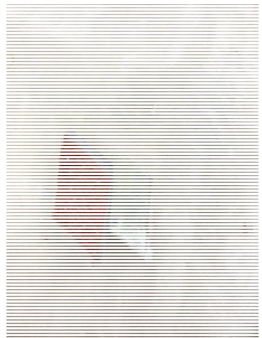


Figure 42: Piece on the left shows adhesion qualities of permanent foaming fixative to substrate, 2nd piece shows no adhesion qualities to substrate

In order to determine what amount of contact the I-R2 foam shall have, 24 samples will be made with some of the samples having a "contaminant" inside acting like a physical barrier from the foam to the substrate. Figure 43 shows the four different scenarios the permanent foaming fixative will be applied in. The "contaminant" will be uniform in thickness and will consist of either a lubricant (grease) or any material that can act as a physical barrier from the permanent foaming fixative to the 304 Stainless Steel substrate.

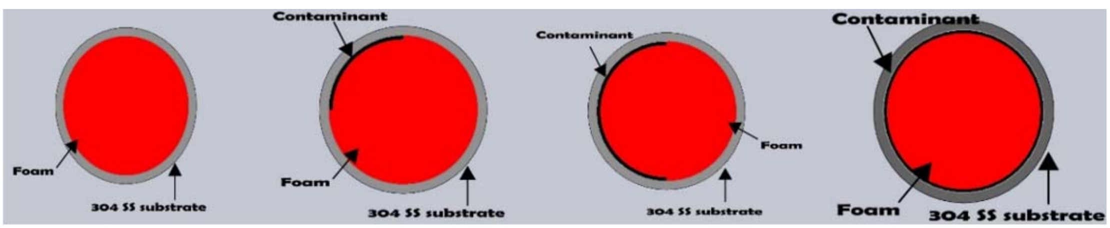


Figure 43: Four testing scenarios: 1st picture on the left shows foaming fixative with no contaminant; 2nd picture shows the foaming fixative with 25% contamination coverage; 3rd picture shows the foaming fixative 50% contamination coverage; last picture shows the foaming fixative will 100% contamination coverage

Section 3.1.5 of ASTM E3191 states:

"Long term measure, n -greater than six months."

same adhesion testing as above will be repeated for similar samples after 1 and 6 months of aging for permanent foaming fixative candidate (I-R2) in the same three operational volumes. The goal is to determine whether time will affect permanent foaming fixatives' immobilizing capabilities.

All findings can potentially update ASTM E3191: *Standard Specification for Permanent Foaming Fixatives Used to Mitigate Spread of Radioactive Contamination* as a guide to conduct mechanical property testing. It is important to note that there are no guidelines to follow to know what amount of stress or loading will cause the release of residual contamination. Overdesigning was performed with this in mind as well as following the same procedures the packaging department at SRNL conducts on polyurethane foams in order to have a prediction of what will happen during an accidental impact.

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7. APPENDIX - EXPERIMENTAL DATA

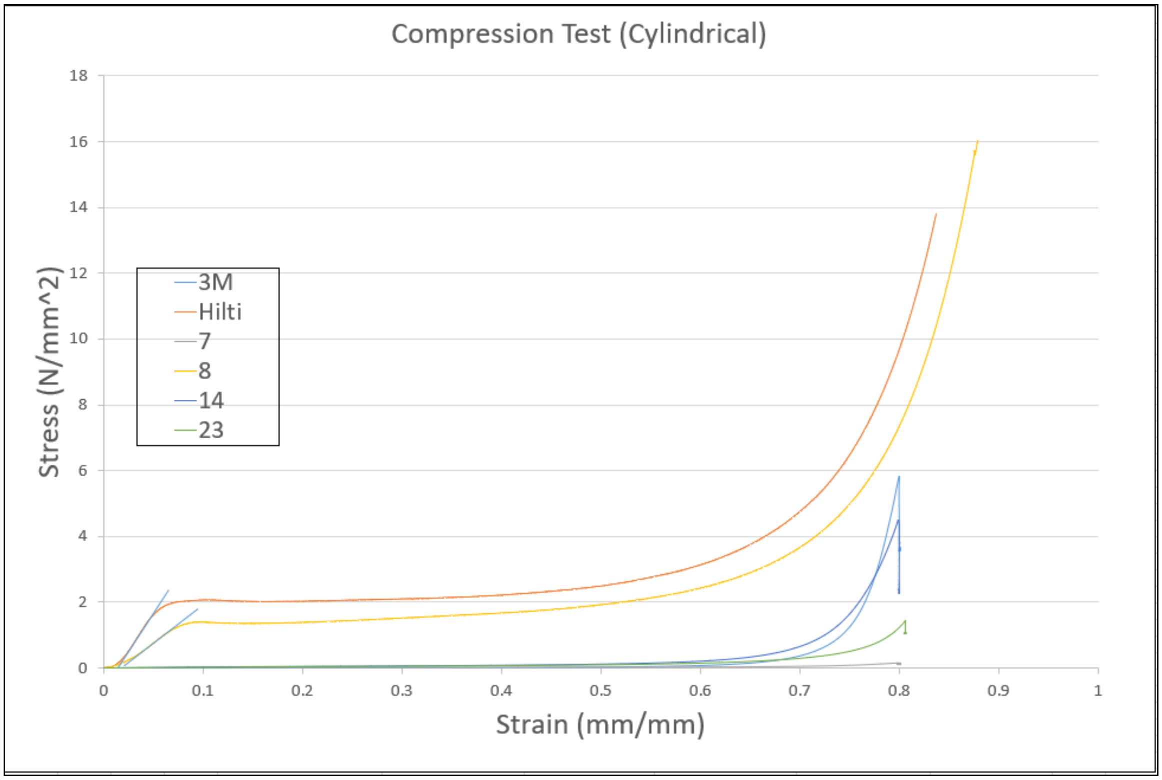

Figure 44: Stress-Strain Graph for all Cylindrical Samples

Table 10: Values from Stress-Strain Graph

Foam Sample	50% Deflection Stress (Mpa)	50% Deflection Load (N)	80% Deflection Stress (MPa)	80% Deflection Load (N)	Thickness Decrease %	Final Thickness (mm)
F-1	0.019	47.83	0.15	389.55	-0.15	20.031
F-2	0.117	293.10	4.39	10994.71	0.14	19.97
F-3	0.095	237.43	1.23	3083.20	13.86	17.22
IF-4	0.026	64.66	5.83	14583.28	11.63	17.67
R-1	1.921	4802.16	7.34	18360.05	71.75	5.65
IR-2	2.487	6218.37	9.68	24213.22	71.76	5.64

Table 11: Stress-Strain Graph for all Rigid Cylindrical Samples

Rigid Foam	Zero-Point Reference	10% Stress (N/mm^2)	10% Load (N)	Modulus (N/mm^2)
8	.01803,.00245	1.37	3415.95	14.51
Hilti	.02,.17	2.05	5125.00	27.09

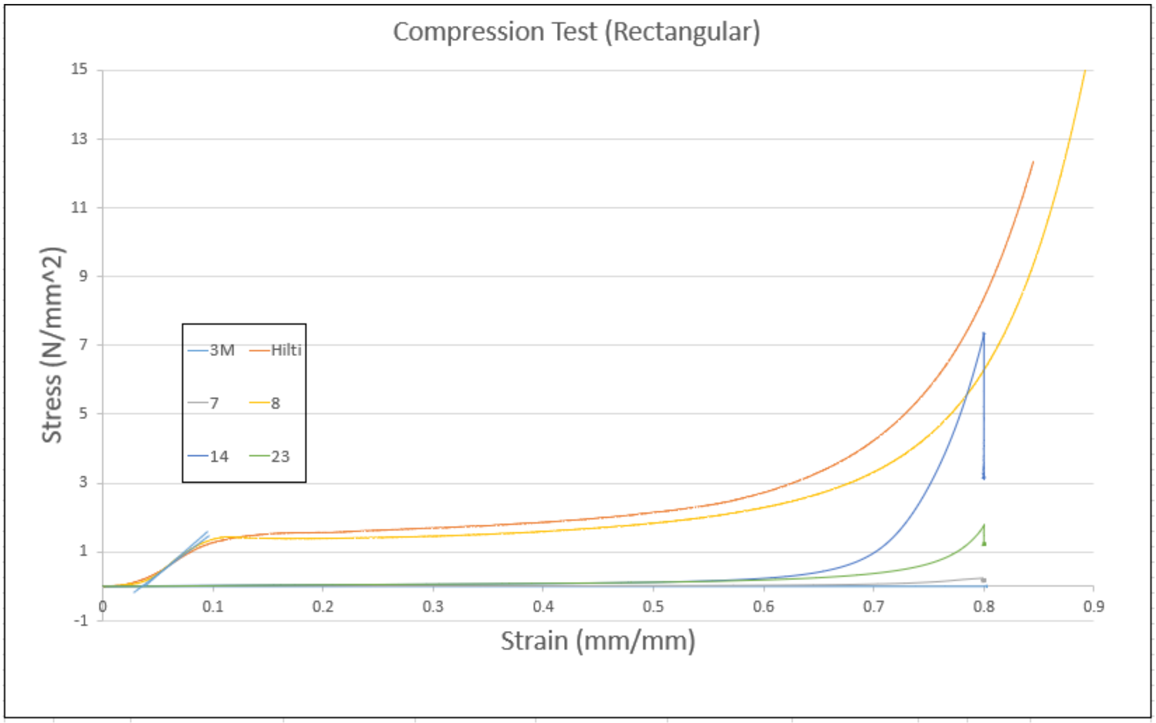

Figure 45: Stress-Strain Graph for Rectangular Samples

Table 12: Values for Rectangular Samples

Foam Sample	50% Deflection Stress (MPa)	50% Deflection Load (N)	80% Deflection Stress (MPa)	80% Deflection Load (N)	Thickness Decrease %	Final Thickness (mm)
F-1	0.0207	51.92	0.24	602.60	1.76	19.64
F-2	0.12	307.76	7.33	18337.82	0.622	19.87
F-3	0.12	304.68	1.77	4447.93	0.335	19.93
IF-4	5.57E-05	0.13	0.00083	2.09	15.5	16.9
R-1	1.83	4587.85	6.28	15708.70	71.22	5.75
IR-2	2.14	5369.12	8.37	20941.27	72.9	5.42

Table 13: Values for Rigid Rectangular Samples

Rigid Foam	Zero-Point Reference	10% Stress (N/mm^2)	10% Load (N)	Modulus (N/mm^2)
8	.01803,.00245	1.42	3550.00	9.76
Hilti	.01357,.125312	1.36	3400.00	10.72