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Evaluation of Canisterized Foams and Evaluation of Radiation Hardened Foams for D&D Activities

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1.0 Executive Summary

The introduction of polyurethane foams has previously been examined elsewhere within the DOE complex with regards to decontamination and decommissioning (D&D) activities, though its use has been prohibited as a result of excessive heat generation and flammability concerns per the safety basis. Should these foams be found compatible with respect to the facility safety basis requirements, D&D work involving large void containing structures such as gloveboxes could be eased through the fixation of residual contamination after decontamination efforts have concluded. To this end, SRNL embarked on a characterization of commercial epoxy foams to identify the characteristics that would be most important to safety basis requirements. Through SRNL's efforts, the performance of commercial two-part epoxy foams was evaluated for their foaming characteristics, temperature profiles, loading capability with high-Z (high density) additives, and applicability for shielding gamma emission from isotopes including; Am-241, Cs-137, and Co-60. It was found that these foams are capable of encapsulation of a desired volume, though the ideal and experimental expansion coefficients were found to differ. While heat is generated during the reaction, no samples generated heat above 70 °C. Of the down–selected materials, heating was on the order of 40 °C for the flexible foam and 60 °C for the rigid foam. Both were found to return to room temperature after 20 minutes regardless of the volume of foam cast. It was also found that the direct introduction of high-Z additives were capable of attenuating 98% of Am-241 gamma signal, 16% of Cs-137 signal, and 9.5% of Co-60 signal at 1:1 loading capacities of total liquid constituent weight to additive weight. These efforts are currently being reviewed for the ASTM January 2017 subcommittee discussions to address the lack of test methods and standards regarding these materials with respect to D&D environments.

2.0 Introduction

Nuclear facilities that are moving towards final disposition, face enormous challenges to ensure no holdup material is released to the environment between the time the facility is no longer active through final disposition operations. Workers actively seek to remove as much of the radioactive materials holdup as possible; however, current decontamination methods are not effective in the removal of all contaminated material. As such, for contaminated areas, there is often some residual contamination remaining after material removal and decontamination efforts have concluded. Gloveboxes are a particular source of hold up contamination that must be addressed, as they are numerous in legacy nuclear facilities.

One method, identified by SRNL, for overcoming these challenges is the use of two-part epoxy foams capable of filling the interior volume of the glovebox to ensure fixation of any remaining material after decontamination efforts have concluded. Previous research by SRNL in this area focused on the introduction of high-Z (high density) additives during the mixing process of these foams to increase the radiological shielding, effectively reducing exposure rates of workers prior to and during the gloveboxes removal or decommissioning. This prior research included modeling additives placed in varied foam constituents to characterize anticipated shielding capabilities using Monte Carlo N-Particle X (MCNP-X) modeling software. This effort proved promising and indicated that over 50% shielding of dose could be achieved with select additives. SRNL also previously characterized five (5) commercially available epoxy foams from the Foam-iT product line (Foam-iT 3, Foam-iT 8, Flex Foam-iT III, Flex Foam-iT 14, and Flex Foam-iT 25) and included the most promising additives for radiological characterization to ensure correlation with the modeled results[1-3]. For the current work, new foams were investigated including 2-part epoxy based fire retardant foams (FlexFoam-iT 7 FR and FlexFoam-iT 23 FR) and compressed fire retardant foams (Abesco FireRated FP200 and Great Stuff Fireblock). More foams have since been received by SRNL and are currently under testing.

3.0 Goals and Objectives

A revised test plan was prepared by SRNL personnel entitled "Radiation Hardened Foam Test Plan – Radiation Shielding Testing and Temperature Profiling, Rev. 1" to structure the testing efforts and ensure the objectives necessary were met by the end of the research period[4]. The goal and objectives of this work are described below.

3.1 Goal

The purpose of this research is to develop and test the efficacy of high-Z loaded fire retardant/resistant foams and characterize their properties with respect to shielding capabilities, thermal effects, and expansion in a closed system should these materials need to be applied in a facility.

3.2 Objectives

Material testing continued through FY17 to further characterize the thermal effects within specific systems, expansion characteristics, off-gas product generation during curing, and shielding capabilities upon addition of additives. Specifically, testing addressed the following objectives:

- Continued investigation of commercial foams and additives
    - Incorporation of other foam manufacturers
    - Testing of fire resistant foams
    - Explore alternative foam materials (e.g. fire retardant foams)
    - Down-selection through established protocols
    - Testing of pressurized foams
    - Development of standardized sample production

Each test allowed for increased narrowing of the ideal foam/additive mixture and for greater radiation characterization of a specific series of foams. Resultant data will enable better advisement of the safety personnel with respect to utilizing this material within a facility.

Initial testing focused on a proof of principle approach and utilized foams procured from the Smooth-On Foam-iT line of products 1. Current research investigated different manufacturer foams including Abesco FireRated FP200 and Dow GREAT STUFF Fireblock and fire retardant foams from the Foam-iT line (7 FR and 23 FR). Also, lifetime testing was performed at the month mark on samples generated in FY16 to ensure no degradation of material.

4.0 Experimental Procedure

4.1 Sample Preparation

All new samples were fabricated for their expansion profiles in a closed environment. SRNL personnel designed and 3D printed a block with removable sides and an interior volume of 2”x2”x2”. Various liquid volumes of epoxy based foams were tested within this mold as well as sprayed volumes of foams from pressurized canisters. First, the mold was assembled with 5 sides and loaded from the top with the foaming material. The top was then securely placed on the mold and the material allowed to cure for 2 hours. After this time, the mold was disassembled and the material extracted for testing and evaluation.

Following cube evaluation, additive testing was completed using the previously established testing protocols to ensure comparability between sample sets. Preparation of loaded and unloaded foam blanks was performed according to manufacturer direction. All foams were received as two-part epoxies that mixed at a given ratio (either 1:1, 1:2, or 2:1). When loading additives, the larger volume material was placed in the container first and mixed thoroughly with the additive, then the second constituent was added and mixed thoroughly for 10 seconds and allowed to foam and cure. Foaming in all instances for small samples was complete within approximately 5 minutes, though full curing per manufacturer direction was allowed to continue for a minimum of 2 hours prior to removal from the mold. The foams chosen all have different mixing profiles and foaming volumes.

4.2 Down-Selection of Foam Materials

The properties of interest for the foam materials include density, expansion rate, and additive capacity. High density foams are favorable as they have a higher gamma absorption cross-section, though the other material properties may prove equally as important in determining the most functional material for D&D scenarios. A survey of common foam epoxies was conducted to evaluate their properties (i.e., expansion rate, density). To complete this task, small scale samples were fabricated by mixing the 2 part foam epoxies and canisterized foams as blanks and with additives to evaluate differences in performance for open and closed volume environments. For these materials, each sample was mixed and allowed to cure fully before total volume expansion was calculated. During the curing process, expansion characteristics were monitored.

4.3 Radiation Shielding Testing (10 Month Evaluation)

Previously down-selected materials from the initial experiments were re-evaluated for their performance at 10 months using gamma sources of varying energy levels. Testing was conducted using sealed Co-60, Cs-137, and Am-241 sources available at SRNL Building 735-2B. To characterize the radiation shielding efficiency of each foam/additive mixture, a FLIR identiFINDER 2 (Figure 1) was used.

Figure 1: FLIR identiFINDER 2

For the initial testing, the identiFINDER was first used to measure the dose rate of the source on contact. A measurement was then taken of the source at a given standoff greater than the thickness of the foam samples (a static 10 cm) to ensure comparable results are obtained across all samples. An initial dose rate was taken for the undoped foam to allow for a baseline shielding percentage to be obtained for the undoped foams to normalize the effects of the introduced additives. Following this measurement, the foam/additive mixture was placed in front of the indentiFINDER with the source remaining at the standoff (10 cm) with the foam and a dose rate measurement was taken (Figure 2). From this information, percent shielding for each foam and source was calculated.

Figure 2: Radiation measurement setup at 10 cm standoff distance (initial setup)

For the 10 month re-evaluation, the setup shown in Figure 3 was used to better control the distance between the source and the detector. This setup utilized 3D printed parts (green) that mounted the source and detector in a fixed orientation and provided a platform for sample loading on-plane with the detection path. This setup proved to have more reproducibility due to the stationary aspects for source and detector and will be used for all subsequent testing of material.

Figure 3: Radiation measurement setup at 10 cm standoff distance (10 month setup)

5.0 Results and Discussion

5.1 <u>Expansion Profiles</u>

To ensure conservation of time and additives, an initial down-selection of the foams was performed based on the expansion profiles of the foam during curing in a closed environment. Initial expansion characterization of each foam was taken upon receipt to ensure that a given liquid volume (~60 mL for this test) foamed to the expected final volume. Figure 4 shows the two epoxy based foams following curing within an enclosed environment. Overall, these materials behaved well with no material failure noted for sufficient initial liquid volume necessary to fill the void volume. Density of these foams was found to be controllable based on initial liquid volume. Characterization of density alteration will be performed in future testing using water displacement methods.

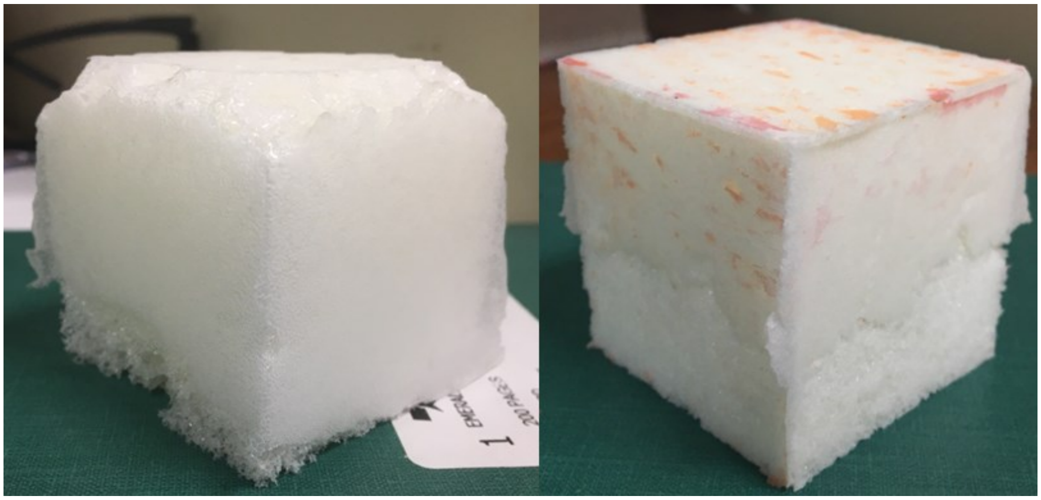

Figure 4: Flex Foam-iT 7 FR (left) and 23 FR (right) [2-part epoxy].

Figure 5 shows the two canisterized foams performance following curing within an enclosed environment. The Abesco sample develops large void spaces that collapse upon curing and disassembly of the mold. Collapsed areas for these materials do not reach full curing, leaving semi-liquid areas within the volume. The Great Stuff material fully cures, however it leaves large void volumes and generates excessive pressure within the confined mold during curing.

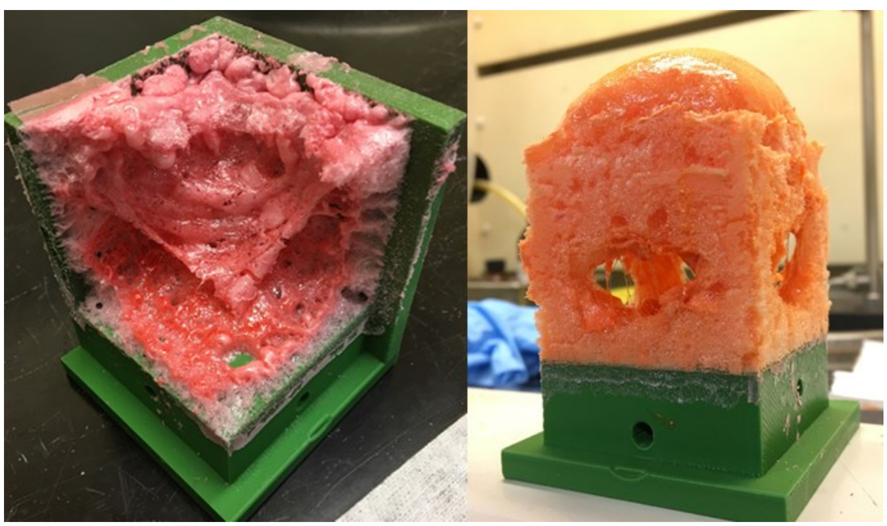

Figure 5: Abesco FireRated FP200 (left) and Great Stuff Fireblock (right) [Canisterized].

5.2 <u>Radiation Shielding</u>

Radiation shielding capabilities were tested for both F8 and F14 loaded foams using three gamma emitting sources of differing energies and dose rates after aging 10 months. Each compound was loaded into foams F8 and F14 at a 0.1, 0.3, 0.5, 0.7, and 1.0 ratio of additive to foam weight. Radiation dose rate measurements were conducted in SRNL Building 735-2B using gamma sources of varying energies. Table 1 provides the initial non-shielding dose readings at a 10 cm standoff distance from the initial and 10 month testing, along with the associated energies of each source. A 10 cm standoff distance (distance from the source to the FLIR identiFINDER 2) was selected to ensure that the differing foam thicknesses did not affect the sources distance from the detector.

Table 1: Sources used for radiation shielding testing

Source	Source ID	Energy (MeV)	Initial Dose at 10 cm (mrem/hr)	Dose at 10 cm 10 month evaluation (mrem/hr)
Am-241	003/11	0.0595	4.94	5.57
Cs-137	F2-785	1.18	15.34	15.19
Co-60	K2-125	2.82	3.78	3.75

Following the radiation measurements, shielding efficiency percentages were calculated. An example of this for both FoamIT 8 and 14 is shown below in Figures 6 and 7 for Am-241. Shielding percent difference (~2-3%) is attributed to error of the device as well as experimental error in measuring the distance of the initial testing which has been remedied through the new fixed experimental setup.

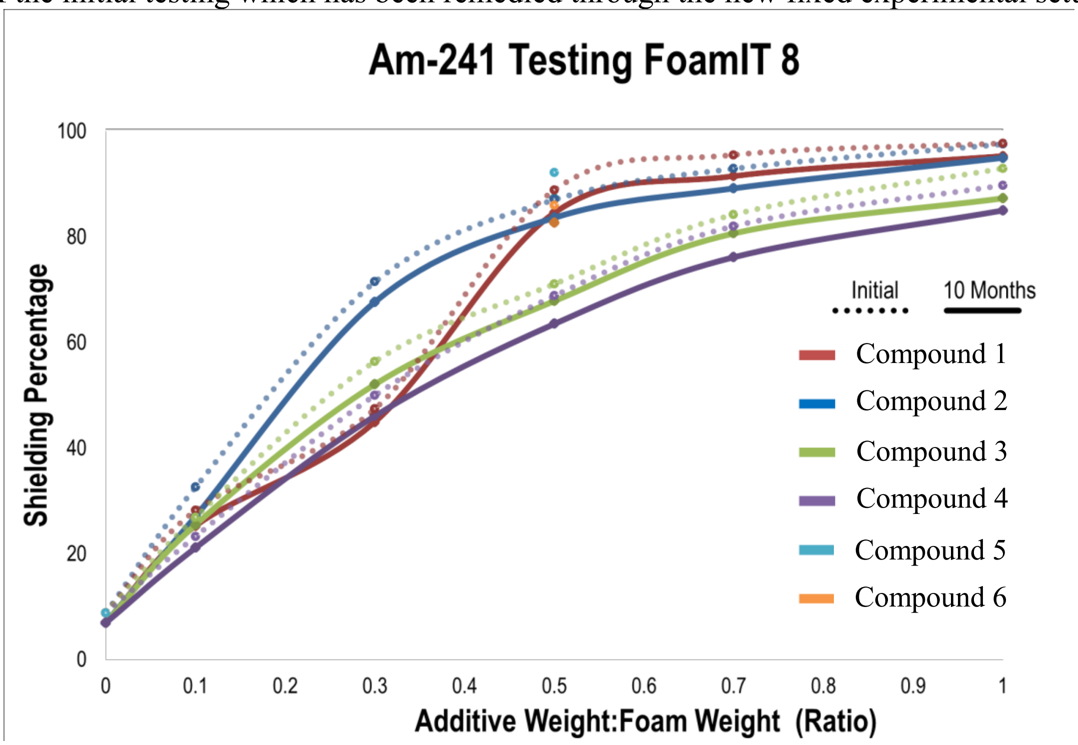

Figure 6: Am-241 Foam-IT 8 Test Data

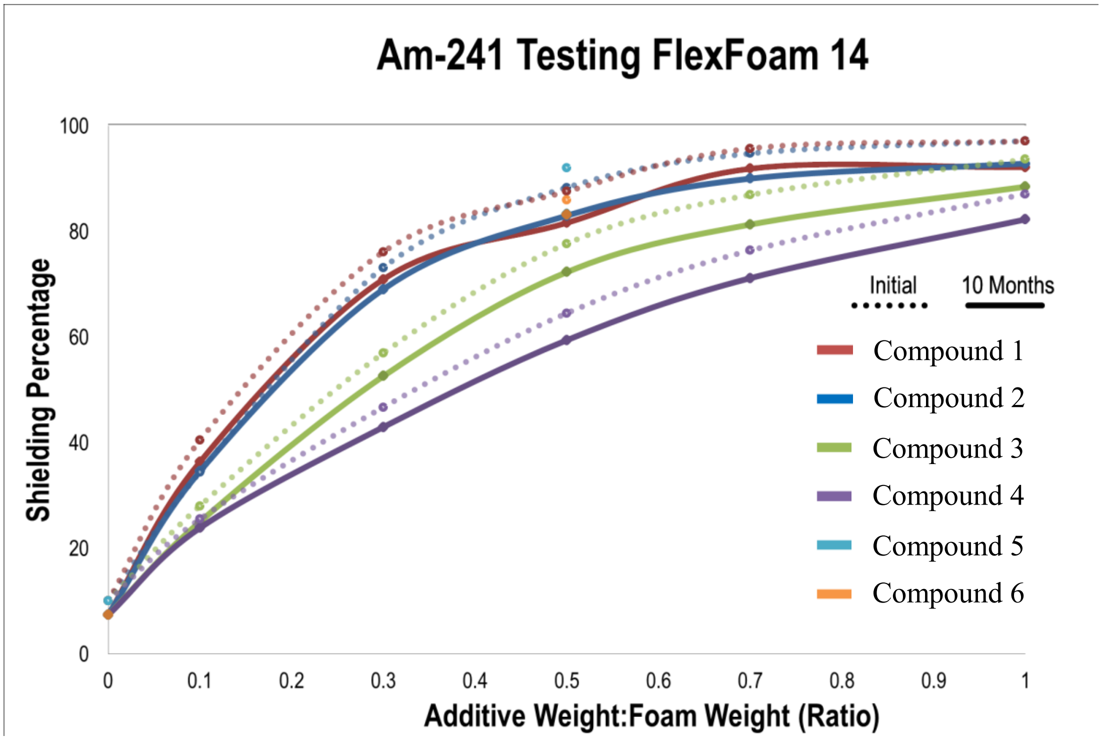

Figure 7: Am-241 Flex Foam 14 Test Data

6.0 Conclusions and Path Forward

Through the experiments above, it was found that the initially developed materials from FY16 were capable of maintaining performance within error of the setup and device over a 10 month period. New materials tested included new fire resistant/retardant epoxy and canisterized foams within enclosed volumes. The epoxy materials were found to perform especially well with the added benefits of 1) ease of additive addition and 2) tailorable densities within enclosed volumes. Canisterized foams in contrast were found to perform poorly for enclosed environments, and failed particularly through the generation of large void spaces that eventually collapse upon themselves or inhibit full curing of the material. These canisterized foams also generate excessive pressure that can potentially rupture the enclosed volume, leading to operational concerns for these materials.

Immediate future work will include further characterization of the epoxy based foams including radiation shielding, density characterization, and additive dispersion throughout manufactured volumes. New materials have also recently been received by SRNL and will be tested per the established protocols above for future material consideration.

This data will be discussed with safety basis personnel present at both SRNL, 235-F PuFF Project and in C Area to aid in the future characterizations that must occur to allow these materials to be introduced into the facility while mitigating any risk per the safety basis. Discussions will also include the possibility of a hot testing for a contaminated glovebox once this technology is more mature to better characterize and understand how the material will behave in-situ. Finally, ASTM standards will be reviewed in January 2018 at the ASTM E10.03 Subcommittee meeting (Subcommittee for Radiological

Protection for Decontamination and Decommissioning of Nuclear Facilities and Components) to determine what ASTM standards and test methods may be modified to address the unique considerations that must be made for these materials and to develop new standards and test methods should no current ones be readily adaptable.

7.0 References

1. *FlexFoam Series III, 14, and 25, Foam iT! 3*; SDS No. 402A; Smooth-On: Macungie, PA, November 24, 2015. https://www.smooth-on.com/msds/files/Flex_Foam-It_Series.pdf.
2. *Foam iT! 8*; SDS No. 470A; Smooth-On: Macungie, PA, December 8, 2016. https://www.smooth-on.com/msds/files/ Foam-It_8.pdf.
3. J. C. Nicholson, B. Peters, J. Wilson, A. L. Washington, II, "Fabrication and Evaluation of Radiation Hardened Polyurethane Foams for D&D Activities," Savannah River National Laboratory. SRNL-L3100-2016-00231 (2016).
4. J. C. Nicholson, B. Peters, "Radiation Hardened Foam Test Plan – Radiation Shielding Testing and Temperature Profiling," Savannah River National Laboratory. SRNL-TR-2016-00311, Rev. 1 (2017).