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ProTec™ Tear-Offs: A Preliminary Assessment

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September 2005

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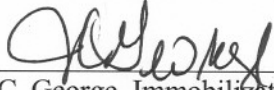
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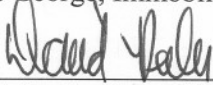


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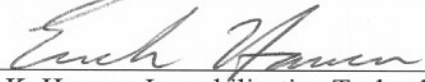


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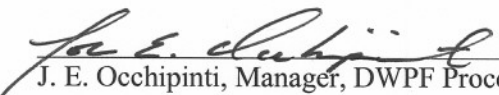
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EXECUTIVE SUMMARY

The Savannah River National Laboratory (SRNL) has conducted a series of “scoping” tests (referred to as Phase 1) to assess the potential use of a Mylar® tear-off system as a primary or secondary protective barrier to minimize acid etching (“frosting”), accidental scratching, and/or radiation damage for shielded cells windows. Conceptually, thin, multi-layered sheets of Mylar (referred to as a “tear-off” system) could be directly applied to the Lexan® sheet or glovebox/hood sash window to serve as a secondary (or primary) barrier. Upon degradation of visual clarity due to accidental scratching, spills/splatters, and/or radiation damage, the outer layer (or sheet) of Mylar could be removed “refreshing” or restoring the view. Due to the multi-layer aspect, the remaining Mylar layers would provide continued protection for the window from potential reoccurrences (which could be immediate or after some extended time period). Although the concept of using a tear-off system as a protective barrier was conceptually enticing, potential technical issues were identified and addressed as part of this Phase 1 feasibility study. These included resistance to:

- (1) acid(s) (concentrated (28.9 M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃),
- (2) base (a simulated sludge with pH of 12.9),
- (3) gamma radiation (cumulative dose of ~200,000 rad), and
- (4) scratch resistance (simulating accidental scratching with the manipulators).

Not only can these four factors play a significant role in determining the visual clarity of the integrated system, they can also contribute to the mechanical integrity issues which could dictate the ability to remove the outer layer when visual clarity has degraded.

The results of the Phase 1 study clearly indicate that the Mylar tear-off concept (as a primary or secondary protective barrier) is a potential technical solution to prevent or retard excessive damage that would result from acid etching, base damage (as a result of a sludge spill or splatter), gamma radiation damage, and/or accidental scratching (due to manipulator/tool contact). The short term tests performed in this task showed that Mylar tear-offs can withstand the chemical and physical abuses expected in off-normal shielded cells operations. The “tear-offs” not only provide some measure of acid resistance, as reflected by the lack of visual degradation after being exposed to four acids, but also act as a protective barrier to accidental contact with the manipulators and/or tools. The conceptual “erasing” of scratches or marks was demonstrated in the shielded cell mock-up facility through the removal, with manipulators, of the outer layer tear-off.

The successful removal of the outer layer tear-off with the manipulator, using tabs not specifically designed for remote operations, demonstrates that the system is “manipulator-friendly” and could be implemented in the shielded cells. The ability to remove the outer layer tear-off not only regains visual clarity but also reduces waste disposal volumes (i.e., disposal of a thin sheet of Mylar which is “collapsible” versus the bulk disposal of a rigid Lexan sheet or glovebox/sash window) which is more cost effective. The “tear-off” system could also reduce the number of cell entries needed to replace the Lexan sheet and increase the time interval between glovebox/sash window change outs which can be costly and time consuming.

Although the primary focus of this report addresses the application of the Mylar tear-offs on shielded cells windows, the concept is also potentially applicable to glovebox and hood sash windows. In fact, the tear-off concept is potentially applicable to any system where visual clarity is compromised given the environmental conditions of the test. In addition, the tear-offs could be applied to walls or shelves where a protective barrier would reduce deterioration or discoloration. This concept is referred to as the ProTec™ tear-off system as its primary intent is to protect windows or surfaces.

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1.0 INTRODUCTION AND BACKGROUND

The Savannah River National Laboratory (SRNL) has conducted a series of scoping tests to assess the potential use of Mylar[®] tear-offs to serve as a primary or secondary protective barrier against acid etching (“frosting”), accidental scratching (e.g., by manipulator or tool contact), accidental spills/splashes (e.g., sludge and/or acids), and/or radiation/contamination damage (α and/or β) for shielded cells windows. The current mitigation technique used in the SRNL shielded cells is to place a rigid, monolithic sheet of Lexan[®] (typically 40” x 36”) in front of the glass window as a protective barrier. Over time, visual clarity of the Lexan deteriorates (due to accidental scratching with the manipulators/tools, spills/splatters, or radiation damage) resulting in the need to replace the Lexan sheet. Historically, SRNL replaces the Lexan sheets in the shielded cells about every 1 – 2 years or on an “as-needed” basis. Replacement is accomplished by cutting a new Lexan sheet to size, retrofitting this sheet with “manipulator friendly handles”, transferring this sheet into the cells, and finally mounting this sheet into place. Subsequently, the Lexan sheet that was replaced is typically size reduced (cut into smaller pieces) and disposed via the appropriate waste disposal route. Similar degradation occurs for glovebox or hood sash windows but in these cases, complete replacement of the window is typically required to regain visual clarity (i.e., generally no “primary” barrier is used). Whether it is the replacement of a large Lexan sheet in the SRNL shielded cells or a direct replacement of a window in a glovebox or hood sash, bulk waste disposal is required which can be time consuming and costly.

The concept being investigated is to use thin, multi-layered sheets of Mylar (referred to as a “tear-off” system) which can be directly applied to the Lexan sheet or glovebox/hood sash window to serve as a secondary (or primary) barrier.¹ Upon degradation of visual clarity due to accidental scratching, spills/splatters, and/or radiation damage, the outer single sheet of Mylar would be removed to refresh or restore the view. If successful, removal of the outer layer would not only regain visual clarity but also provide the “next layer of protection” due to the multi-layered concept. A significant advantage of this system is that it reduces the waste disposal volume (i.e., disposal of a thin sheet of Mylar which is “collapsible” versus the bulk disposal of a rigid Lexan sheet or glovebox/sash window – which may require size reduction to fit disposal containment). If successful, the tear-off system would also reduce the number of cell entries needed to replace the Lexan and increase the time interval between glovebox/sash window replacements which are costly and time consuming.

Although the concept of using a tear-off system as a protective barrier is conceptually enticing, there are potential technical issues or show-stoppers that need to be addressed. Based on a literature review of the Mylar product coupled with the potential applications, the primary technical issues that must be addressed to demonstrate feasibility include:

- acid/base resistance (e.g., against an acid or sludge spill/splatter),
- scratch resistance (e.g., against accidental contact of the window with the manipulator and/or tool),
- radiation damage resistance (e.g., against radioactive sources in shielded cells and/or gloveboxes), and
- mechanical integrity after acid/base exposure, scratching, and/or radiation damage (i.e., would the tear-offs lose mechanical strength to the extent that the outer layer tear-off could not be completely removed?).

¹ The multi-layer Mylar tear-offs could be mounted directly onto the high level caves windows as the primary barrier – replacing the Lexan. However, until adequate technical data are in-hand the authors would strongly encourage that the tear-off system be used as a secondary (or back-up) system in this and/or other service conditions. It should be noted that terminology of secondary or primary barrier does not include the glass which also serves as a containment system.

A successful evaluation of the primary technical issues would demonstrate feasibility of the concept as well as provide insight into a more detailed study of other potential scenarios that would challenge or test the use of the Mylar tear-offs under realistic or potentially bounding service conditions.

It should be noted that the “tear-offs” used in this testing were an “off-the-shelf” system designed for a high-speed racing application (more specifically, tear-offs designed for motorcycle helmets).² For its intended use, the multi-layered tear-off system is applied to the visor of the helmet and as visual clarity degrades during the race (due to sand blasting, oil, fuel, mud, rubber, or water coverage), the rider simply reaches for a tab on the outer tear-off and removes it. This not only refreshes his/her view but the multi-layer aspect of the system provides additional protection for the potential reoccurrence (which could be immediate or after some extended time period). Given the tear-offs being used are not specifically designed for the desired application (e.g., shielded cells, glovebox, or hood sash windows) compositional changes may be warranted to enhance the resistance to either acid exposure, scratching, and/or radiation damage – if the “off-the-shelf” technology proves ineffective under the simulated test conditions of this study or other service conditions which have not been evaluated.

Although the primary focus in this report addresses the application of the Mylar tear-offs on shielded cells windows, the concept is also potentially applicable to glovebox and hood sash windows. In fact, the tear-off concept is potentially applicable to any system where visual clarity is compromised given the environmental conditions of the test. In addition, the tear-offs could be applied to walls or shelves where a protective barrier would reduce deterioration or discoloration (e.g., the walls of a chemical hood).

2.0 OBJECTIVE

The objective of this study is to demonstrate the feasibility of the Mylar tear-off system to serve as an effective primary or secondary barrier against accidental scratching and/or damage incurred as a result of radiation or acidic/basic reactions. To meet this programmatic objective, a phased approach is being used. In Phase 1, three primary objectives were addressed which include assessments of: (1) acid resistance, (2) base resistance, and (3) scratch resistance.³ In addition, an in-situ cell demonstration was initiated to gain some insight into the behavior of the system under more realistic service conditions in terms of gamma radiation damage (to assess the potential impact of gamma radiation on the optical and mechanical properties of Mylar).

The Phase 1 tests outlined above were simple to conduct and relatively inexpensive given the use of “go-no-go” acceptance criteria (discussed in Section 3.0) without detailed analysis being required. The “go-no-go” acceptance criteria are based primarily on visual clarity and simple mechanical integrity tests (i.e., the ability to completely remove the outer layer tear-off after some degree of exposure). Although the

²The tear-offs used were obtained from Racing Optics (San Clemente, CA). Technical information (albeit limited) was obtained on a DuPont Mylar product similar to the multi-layer tear-off product to be used in this testing. The technical data indicated that the chemical, electrical, optical, and physical – thermal properties of Mylar vary as a function of test or environmental service conditions. For example, mechanical degradation data (e.g., tensile strength) were presented for various acid molarities (including HCl and HNO₃) and exposure times. In general the data suggested that as the acid molarity increased, the loss of mechanical strength increased (which could have an impact on the ability to remove the outer layer tear-off). The data associated with optical integrity suggested that “hazing” occurred upon exposure to various acids and bases. Although degradation of mechanical integrity and/or optical quality was noted, the standard testing performed to obtain this information was based on immersion tests – not typical (not even an extreme condition) for the planned application. Based on this information and recognizing the relatively aggressive test conditions under which it was obtained, Mylar should provide at least similar (if not more) protection as compared to Lexan.

³The use of “scratch resistance” is a misnomer given the Mylar is susceptible to scratching. The purpose of the Mylar is to prevent damage to the cell, glovebox, or sash window to the extent that upon removal of the outer tear-off the “scratches or marks” would be removed resulting in an unimpeded view.

Phase 1 tests will provide insight into the potential application of the tear-offs to meet the stated objectives, interactive tests (i.e., a tear-off system exposed to a combination of an acid/base environment, induced scratching, and/or radiation damage followed by an assessment of mechanical integrity) should be performed prior to implementation – Phase 2. However, if show-stoppers are observed in Phase 1, there would be no value in conducting the more time consuming and costly Phase 2 tests.

It should be noted that the duration of the feasibility tests and their termination times were somewhat subjective. Obviously, the longer the Phase 1 exposure times, the more confident one becomes in the system assuming no critical failures are encountered. The primary reason for terminating a specific test “early” was to assess the mechanical integrity of the tear-offs after exposure to certain test or service conditions to support the determination of feasibility for this report. For example (and as will be discussed), removal of the outer layer tear-off in the acid exposure tests was performed prior to visual degradation to ascertain mechanical integrity issues. Given the multi-layer aspect of the tear-off system, the acid resistance test were “restarted” immediately to assess longer term effects but the exposure time was reset to zero.

3.0 METRICS TO MEASURE “SUCCESS”

Although this task is scoping in nature, there is a critical decision to be made based on the Phase 1 results. That is, should additional testing be performed to assess the viability of the “tear-off” concept for actual implementation? If so, what should that testing consist of? The primary inputs to that decision making process are the metrics by which the tear-offs are judged in relation to meeting relatively ill-defined success criteria. The two primary metrics for success used during Phase 1 were: (1) visual clarity and (2) mechanical integrity. The latter metric (mechanical integrity) is rather straight forward in terms of classifying success. More specifically, if the outer (or top layer) tear-off can not be completely removed by the use of manipulators (or by hand in other applications such as glovebox or hood sash use) then the system or concept is of no value. Complete removal of the tear-off after exposure to acids, bases, physical impact, radiation or any combination is mandatory. More specifically, even if visual clarity was not reduced, if mechanical integrity deteriorates to the point where it results in the incomplete removal of the tear-off (i.e., ripping), that would be unacceptable from a practical application.

With respect to visual clarity, the success metric becomes a little more ill-defined. That is, documentation via notebook entries and time-lapsed digital photos provides a measure of success – but what level of visual degradation would result in a decision to terminate the possible implementation of the concept? The answer may reside not in how much visual degradation occurs, but in how long does it take to reach some critical or detrimental level? Current practice in the high level caves requires changing out the Lexan sheet on some time interval basis (typically 1 – 2 years). If the tear-offs extend this time interval, one may classify this as a “success.” In judging this potential, one also has to remember that the tear-offs are multi-layered so visual degradation success should perhaps be judged on the integrated time the tear-off system provides access to visual clarity relatively to the 1- to 2-year time period typically observed for each Lexan plate.

How long is long enough? Obviously, the Phase 1 data do not span multiple years but have been designed to provide a fair assessment of feasibility under potentially bounding service conditions. The technical issues and potential for implementation may ultimately be resolved by comparing “longer-term” tests to historical experience by the operators of the intended application. However, the Phase 1 testing provided some confidence in carrying the concept forward although it only spans a relatively short duration (~2.5 months).

4.0 EXPERIMENTAL APPROACH

As previously mentioned, the Phase 1 testing evaluated acid, base, and scratch resistance as well as an in-situ test where a small scale version of the Lexan – “tear-off” system was monitored in Cell #15 of the SRNL shielded cells. A more detailed discussion of each Phase 1 test is provided below.

4.1 Acid Resistance

To support this assessment, four sets of 4 mil (3 layer) Mylar tear-offs were mounted to separate Lexan plates (Lexan sheets that were cut to fit the tear-offs used in this test) and suspended 4” above vessels containing four different acids. The use of 4” was essentially a compromise between two potential bounding service conditions: (1) placing the tear-offs directly onto the acid baths (initially considered to be overly aggressive – not representing an actual service condition) and (2) some larger distance (> 4”) which may not be aggressive enough. As will be discussed, after 13 days at 4”, all of the plates (with and without tear-offs) were dropped from 4” to 2” due to concerns of hood air flow and the potential impact of reducing exposure to the tear-offs. Ultimately, the tear-offs were placed directly on the vessels holding the acids with no spatial gap (referred to as a 0” level).

Figure 4-1 shows a schematic of the acid resistance tests. The acids and concentrations used were: concentrated (28.9M) HF, concentrated (15.9M) HNO₃, 6M HCl, and 0.6M H₃BO₃.⁴ Teflon vessels having a capacity of 250 mL were filled with approximately 200 mL of acid. In addition to the “tear-off” (Lexan + tear-off system), a “blank” (Lexan-only) was also suspended (initially at 4”) above each acid bath as a reference (simulating the SRNL shielded cells use as the primary barrier). The Lexan blanks provide a baseline or measure of how aggressive the specific test conditions (acid type, acid molarity, and/or suspended height) were with respect to historical observations in the SRNL shielded cells. For example, a test condition in which the Lexan blank begins to lose visual clarity within a short-time frame may be indicative of an overly aggressive condition that has not been observed in actual field situations. Additionally, the blanks provide a basis for each test condition when comparing the blank with that of the Lexan + tear-off system.

⁴ The acids and molarities were specified by the Defense Waste Processing Facility (DWPF) Analytical Laboratory who supported this particular work scope.

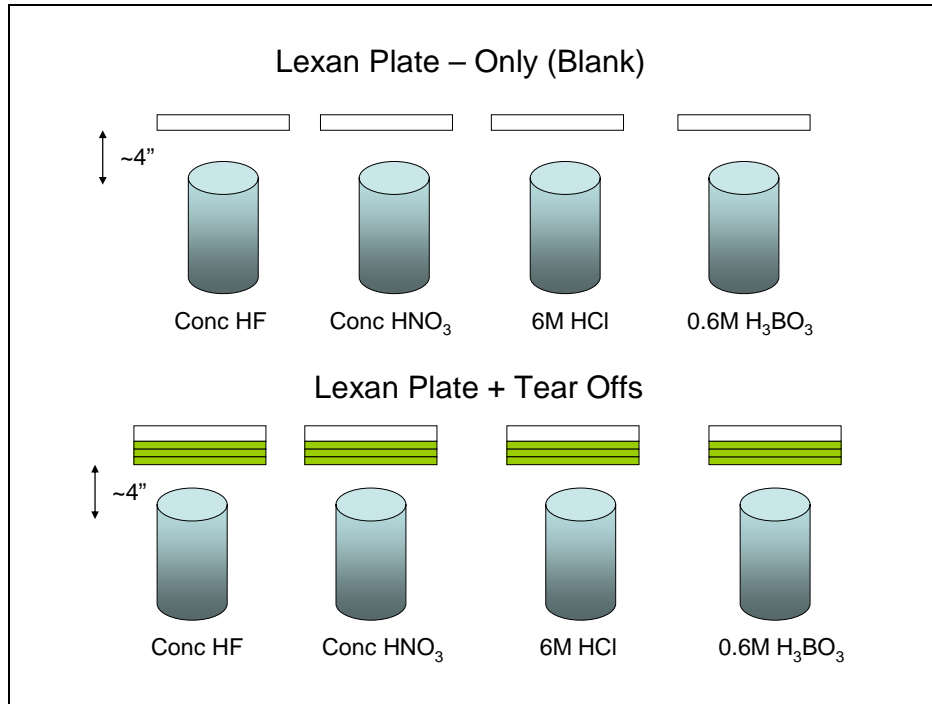


Figure 4-1. Schematic of Acid Resistance Testing Initial Set-Up

A visual baseline condition (pre-exposed) of the “blank” and “tear-off” systems was required prior to starting the actual tests. This was performed by taking a digital photo (see Figure 4-2) of a pre-exposed Lexan “blank” (on the left side of the clock) and “tear-off” system mounted on the Lexan (on right side of the clock). The “wall clock” was used as a back-drop to provide a basis for visual clarity (color and sharpness of lettering). Although hard to distinguish via the digital photos, the “tear-off” provided a slight tinting of the white background of the clock as compared to the baseline “blank”. There was no reduction to the sharpness of the lettering.



Figure 4-2. Pre-Exposed Condition of the Lexan “Blank” (left) and “Tear-off” System (right)

In general, the “tear-offs” and “blanks” were inspected daily for physical degradation and/or visual distortion and photographed periodically for documentation/comparison purposes.⁵ As the system was monitored over the first few weeks, observations were made which resulted in changes to this initial test conditions. These observations and resulting physical changes to the set-up are discussed in more detail in Section 5.0.

⁵ All visual and recorded information regarding the Phase 1 tests are documented in WSRC-NB-2004-00136.

4.2 Base Resistance

One of the scenarios of particular interest was to assess the ability of the tear-offs to tolerate an accidental spill or splash of a DWPF sludge sample which are basic in nature (i.e., pH values of 10 or greater). To address this issue, a simulated DWPF sludge (produced at SRNL) was placed on a Lexan “blank” and a “tear-off” system using a pipette. Figure 4-3 shows the set-up of this test.⁶ The simulated sludge had a measured pH of 12.9.⁷ After 24 hours of contact, the sludge was wiped from both the “blank” and the “tear-off” and visual observations were documented. Mechanical integrity of the tear-off was monitored via removing the outer layer tear-off. It should be noted, if an accidental spill or splash of a sludge did occur in the shielded cells facility, the sludge would be wiped off immediately – thus the 24 hours was a reasonable bounding service condition or simulated an accidental spill or splash.

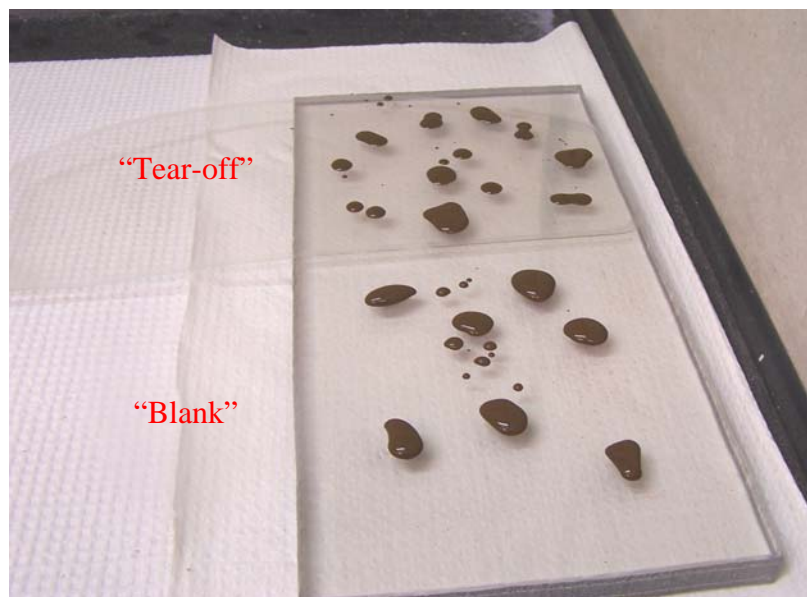


Figure 4-3. “Base” Resistance Test Set-Up: Lexan “Blank” and Mylar Tear-Off System
(photo taken on August 31, 2005 – just after test was initiated)⁸

4.3 Scratch Resistance

The “off-the-shelf” tear-off technology being used was designed to minimize abrasion damage from high speed impacts – the potential application in the shielded cells exposes the system to different risks. The primary physical damage to the tear-offs in the shielded cells is from accidental scratching by the manipulators (or other materials such as a tool or vessel being held by the manipulator) as they contact the cell window. As previously noted, the use of “scratch resistance” is somewhat of a misnomer given the Mylar is susceptible to scratching. The purpose of the Mylar tear-off is to prevent damage to the underlying Lexan sheet, glovebox, or sash window to the extent that upon removal of the outer tear-off,

⁶ Due to the limited supply of tear-offs, the system available to support this test had the Mylar tear-off mounted in such a manner that approximately ½ of the tear-off system was on the Lexan plate while the other half extended over the edge (see Figure 4-3). Although inconsistent with the intended application, this will allow for an assessment of the impact or interaction of a high pH material with both the Lexan “blank” and Mylar tear-offs.

⁷ The simulated sludge used was FPMR-0136 (a Sludge Batch 3 simulant) produced using a co-precipitation process.

⁸ To provide a frame of reference, the Lexan plate is roughly 5” (w) x 10” (l).

the “scratches or marks” would be removed (conceptually erasing or clearing any visual obstructions) while providing the next layer of protection.

To address this issue, a Lexan “blank” and a “tear-off” system were placed into the SRNL shielded cells mock-up facility. Prior to testing, visual clarity of the Lexan “blank” and “tear-off” system was documented (photographed) for comparison purposes, see Figure 4-4. A single sheet of Lexan (~ 10” x 10”) was used with a multi-layered “tear-off” system mounted on one side of the Lexan. The Mylar tear-off can be seen on the right hand side of the Lexan plate (in Figure 4-4) and is approximately 9.5” (high) by 4.5” (at its widest or mid point). To assess visual clarity, laminated “test patterns” were placed behind the monolithic Lexan plate – one behind the Lexan “blank” while the other test pattern was placed directly behind the “tear-off” system.

It should be noted that the “off-the-shelf” tear-offs have individual (but small) tabs located on a corner, which permits the easy removal of the sheets by hand. However, these tabs were not designed for the manipulators and thus modifications were made to the tabs to allow the manipulators to grab the tab more readily.⁹ A “modified” tab (lower right in Figure 4-4) was placed on the three individual tear-off tabs which permits the manipulators to grab the outer layer tear-off to assess removal potential after physical damage had been induced.

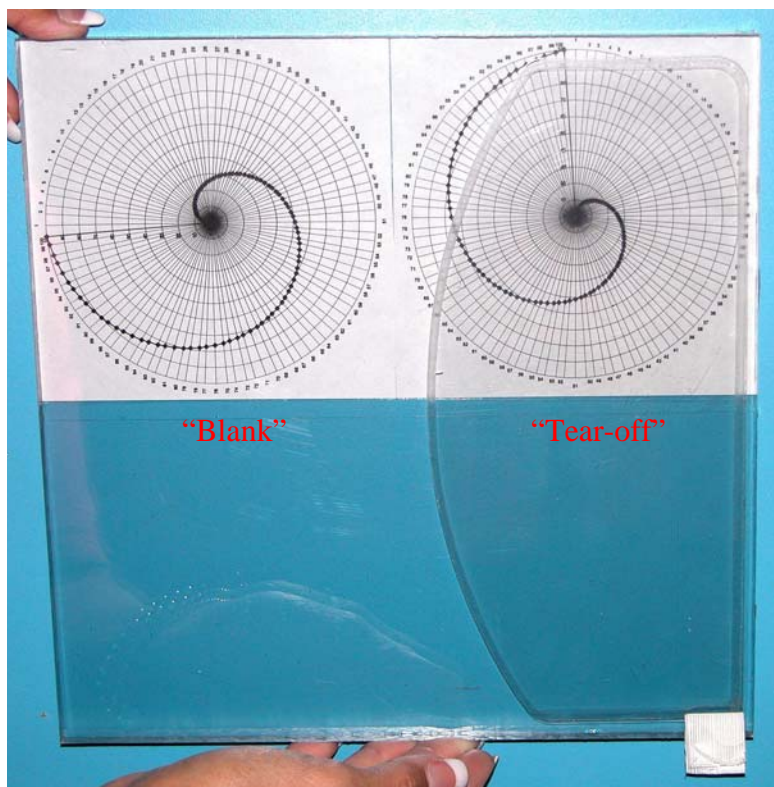


Figure 4-4. Lexan Plate with Mounted Tear-Off Prior to Scratch Resistance Testing

⁹ If this concept were to be implemented in the field as a protective barrier, the tabs would have to be modified with manipulator handling in mind and potentially built into the design and manufacturing process.

The plate was mounted in mock-up facility. The manipulators were then used to test the scratch resistance via simulating the accidental scraping/scratching of the windows. With respect to the physical nature of the tests (i.e., the magnitude and frequency of manipulator contact with the “blank” or “tear-offs”), there were no “official” guidelines or procedures to govern this effort – given contact is accidental. That is, to what degree should the shielded cells technician “abuse” the tear-offs with the manipulator to assess scratch resistance for the Phase 1 tests? Given no set condition, the test was performed using what was considered to be a bounding approach from the shielded cells personnel perspective. That is, the degree or severity of interaction was rather excessive to simulate a long-term service or exposure which would yield multiple contacts. For example, the shielded cells technician intentionally induced on the surfaces of both the “blank” and “tear-off” systems using a force and/or frequency that were inconsistent with normal manipulator operations in the shielded cells. The manipulator was used as both a hammer, beating the “blank” and “tear-off” systems and as a scraper, running the manipulator grips up-and-down the face of the Lexan sheet transitioning back-and-forth between the “blank” to the “tear-off” systems inducing marks/scrapes along the way. Again, the primary interest was the mechanical integrity of the tear-off system after inducing the scratches and the ability to “erase” the visual degradation upon removal of the outer layer.

4.4 In-Situ Cell #15 Test

The in-situ cell demonstration was not specifically designed as a bounding assessment, but one that provides insight into the potential use of the Mylar tear-off under realistic test conditions when considering the application in a high radiation environment. Selection of Cell #15 was primarily based on two factors: the high gamma radiation background (Bibler (2005) reported an average gamma radiation dose of 17.3 rad/hr for Cell #15) and the absence of a heat source. Selection due to the high radiation background is obvious while the lack of a heat source eliminates any possible combustion issues.¹⁰ It should be noted that the tear-offs will not be effective in protecting the Lexan plate against gamma radiation (given the high penetration depths), but could be effective in mitigating alpha contamination damage and/or beta radiation damage for other service conditions. However, the gamma radiation may induce embrittlement which may negatively impact mechanical integrity and the ability to completely remove the outer layer tear-off.

The in-situ Cell #15 test used a single sheet of Lexan with a Mylar tear-off system mounted on one side, similar to the mock-up scratch test described in Section 4.3. To assess visual clarity as a function of time, laminated test patterns were placed behind the Lexan sheet – one behind the “blank” system and another directly behind the “tear-off” system.¹¹ Figure 4-5 is the baseline condition of the Lexan sheet prior to Cell #15 entry. As previously noted, the “tear-off” provides a slight tinting effect, which was observed in Figure 4-2 and Figure 4-4.

¹⁰ Combustion issues were a concern given the unpredictability of the physical integrity of the tear-offs. More specifically, the tear-off layers are held together by either “glue” or through static compression. Assuming the high radiation background resulted in the sheets separating and falling, heat sources could result in combustion – an unacceptable situation for the feasibility tests and a service condition or environment that needs to be addressed if implementation is considered.

¹¹ To provide a frame of reference, the Lexan plate is roughly 10” x 10”. The tear-offs (mounted on the right hand side) are roughly 9.5’ long and ~4.5” wide at the maximum width (center or mid-point).

The Lexan sheet was placed in Cell #15 on July 28, 2005 and was monitored for visual clarity on a routine basis. On September 12, 2005 (~46 days of exposure) the outer layer tear-off was removed with the manipulators to assess both mechanical integrity and ease of removal with the manipulators.¹² The Lexan sheet remains in Cell #15 (with 2 tear-offs remaining) for longer-term monitoring.



Figure 4-5. Baseline of “Blank” and “Tear-Off” System Prior to Cell #15 Entry
(Lexan “blank” on left; mounted “tear-off” on right)

¹² Although the removal of the tear-offs does provide insight into feasibility, the size and design of the tear-off tabs should be designed into the fabrication process to ensure complete removal under actual conditions.

5.0 RESULTS

In this section, the Phase 1 results are presented. As previously noted, the termination of a specific test was based on the need to obtain data for the feasibility study and was not based on the degradation of visual clarity or mechanical integrity. More specifically, under certain test conditions, the outer layer tear-off was removed to assess mechanical integrity prior to any noticeable degradation in visual clarity in order for the results to be documented in this report. Obviously, longer exposure times would provide more confidence, for the tear-offs, assuming no critical failures are encountered. Even though the outer layer was removed, the multi-layer aspect of the system allows long-term monitoring under specific test conditions.

Tables 5-1 through 5-3 summarize the major events (in chronological sequence) for the acid resistance, base resistance, and shielded cells tasks, respectively. Along with the major events, high level summary comments are also provided. The tables are presented at this point as a reference guide for the upcoming discussions.

Table 5-1. Summary of Acid Resistance Testing

Event/Observations	Date	Comment
Initiated Acid Resistance Testing at 4" Level	June 29, 2005	-
Corrosion noted on HCl and HNO ₃ steel supports	July 11, 2005	No signs of visual distortion of "tear-offs" or "blanks".
Decision to lower Lexan plates from 4" to 2" above acid	July 12, 2005	13 days into testing, no signs of visual degradation of "tear-offs" or "blanks".
Performed litmus paper test to ensure acid vapor – Lexan interaction	July 12, 2005	Results confirmed vapors contacting "blanks" and "tear-offs".
Conc. HNO ₃ splash due to mechanical failure of ring stand	July 27, 2005	Droplets of HNO ₃ noticeable on outer layer tear-off, provides insight into realistic scenario of accidental acid splash. No signs of visual degradation of "tear-offs" or "blanks".
Removal of outer layer tear-off from acid tests	August 8, 2005	39 days total exposure (at varying heights), no signs of visual degradation, complete removal of outer layer tear-off from each acid without "ripping" or partial removal.
Moved "blanks" and "tear-offs" to the 0" level for the acid resistance test	August 12, 2005	The 0" level represents essentially no physical gap between the acid bath and the blank or tear-off. This is an aggressive set-up in terms of actual service conditions.
Yellow circular stain noted on "blank" under the HNO ₃ acid tests	August 25, 2005	After 17 days at the 0" level, no signs of "yellow" circular stain on "tear-off" system, all other systems were visually clear.
Completion of Phase 1 Acid Resistance Testing	September 12, 2005	35 days total exposure at 0" height (2 nd layer tear-off), no visual signs of degradation, outer layer tear-offs were not removed.

Table 5-2. Summary of Base Resistance Testing

Event/Observations	Date	Comment
Initiate testing using DWPF simulated sludge	August 31, 2005	-
Terminated testing	September 1, 2005	~24 hours exposure to pH 12.9 simulated sludge, no visual damage to tear-off, outer layer successfully removed to restore visual clarity.

Table 5-3. Summary of Shielded Cell Testing

Event/Observations	Date	Comment
Lexan tear-off system placed in SRNL Shielded Cells (Cell #15)	July 28, 2005	-
Performed “scratch resistance” testing in shielded cells mock-up facility	August 25, 2005	Very aggressive contact between tear-off system and manipulator, marks/scratches induced on tear-off and Lexan blank, removed outer layer tear-off without ripping, visual clarity restored.
Completion of Phase 1 In-Situ Cell #15 Testing	September 12, 2005	46 days of in-situ testing, no visual signs of degradation, complete removal of tear-off with manipulator.

5.1 Acid Resistance

During the early stages of monitoring the acid resistance tests, a few technical issues or concerns arose associated with the actual set-up equipment; not the “blank” or tear off systems. The primary issues were the impact the test setup had on the minimum hood air flow requirements, Lexan plate height (4”), and corrosion of the stainless steel support system observed on some of the acid ring stands. More specifically, after 13 days of exposure at the 4” mark, deposits or corrosion was observed on the stainless steel support system or rings holding the acid vessels for the concentrated HCl and to a lesser degree for the concentrated HNO₃. Figure 5-1 through Figure 5-4 not only show the “corrosion” issue (on select test support systems) but also provide an initial assessment of visual clarity of each system after 13 days of exposure to each acid at a 4” plate height. Figure 5-2 shows the corrosion of the ring stand for the concentrated HCl bath. The digital photos were taken looking down through the Lexan blanks (left) and/or tear-off system (right) into the acid baths. A measure of visual clarity can be obtained by the sharpness of the letters on the acid vessels. These photos can be compared to the baseline condition shown in Figure 4-2.

The corrosion is indicative of acid vapors being carried toward the back of the hood and/or down the sides of the acid vessels – potentially minimizing the amount of acid reaching the “blank” and tear-off systems. To address this issue, litmus paper was mounted on the underside of the “blank” and tear-off over the concentrated HF vessels and the results (i.e., paper turned pink color) suggested that vapors were interacting with the systems at the 4” position. However, a decision was made to lower the systems to 2” to hopefully allow for a more intimate contact of the vapors. After physically altering the set-up, a series of litmus paper strips was mounted to the underside of the Lexan plate over the 6M HCl vessel. After

only a few minutes of exposure, a color change was noted, indicative of acidic vapor interaction. Over time the litmus paper transitioned from “green” (pH ~ 6, when wet with water) to “pink” indicating a pH of ~ 2. Figure 5-5 shows the litmus paper after 24 hours of exposure at the 2” level.¹³

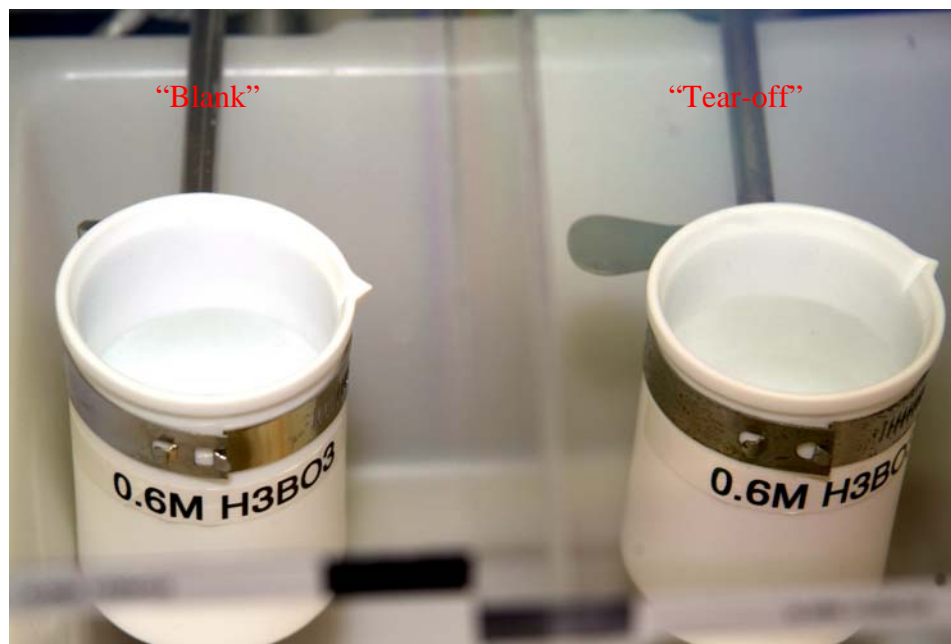


Figure 5-1. Visual Clarity of “Blank” (left) and “Tear-Off” (right) after 13 Days (0.6M H₃BO₃)
(plate heights are 4” above the acid vessel)

¹³ After repositioning from 4” to 2”, “brown” deposits were observed on the top surface of the plate (opposite to the surface exposed to the acid) and on the edge of the plate (near the front of the hood) – presumably a result of contamination from handling. These deposits were documented in WSRC-NB-2004-00136. It should also be noted that the humidity level in the glovebox containing the HNO₃ tests was extremely high during the 2.5 month Phase 1 test period. As a result, some observations documented in WSRC-NB-2004-00136 refer to “residual water” or condensation being observed on the inside of the hood sash window as well as potentially covering the Lexan plates.

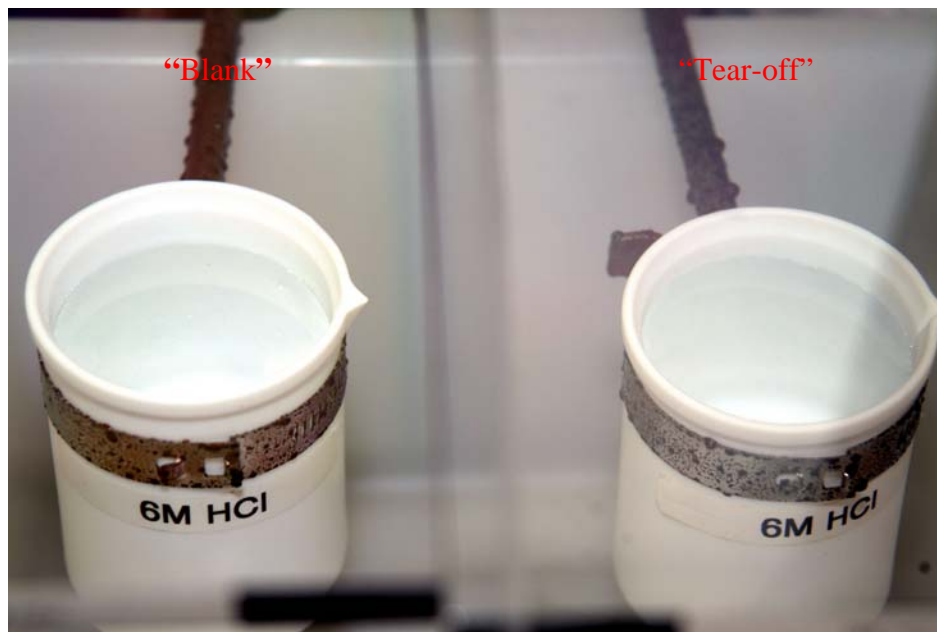


Figure 5-2. Visual Clarity of "Blank" (left) and "Tear-Off" (right) after 13 Days (6M HCl)¹⁴
(plate heights are 4" above the acid vessel)



Figure 5-3. Visual Clarity of "Blank" (left) and "Tear-Off" (right) after 13 Days (Concentrated HF)
(plate heights are 4" above the acid vessel)

¹⁴ Note corrosion / deposits on the metal rings and rods holding the 6M HCl acid vessels.

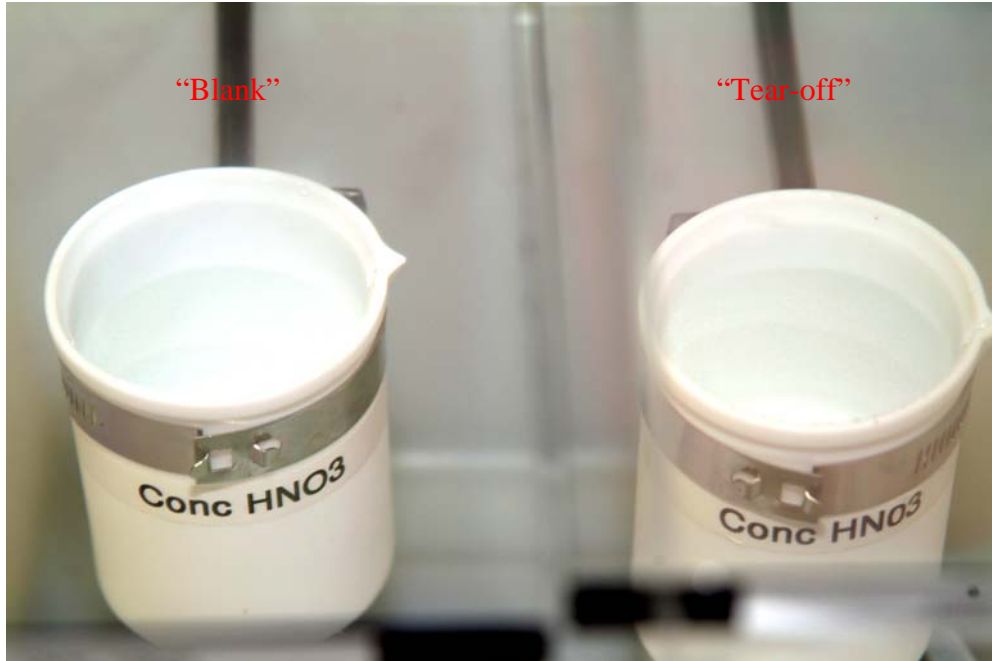


Figure 5-4. Visual Clarity of “Blank” (left) and “Tear-Off” (right) after 13 Days (Concentrated HNO₃) (plate heights are 4” above the acid vessel)

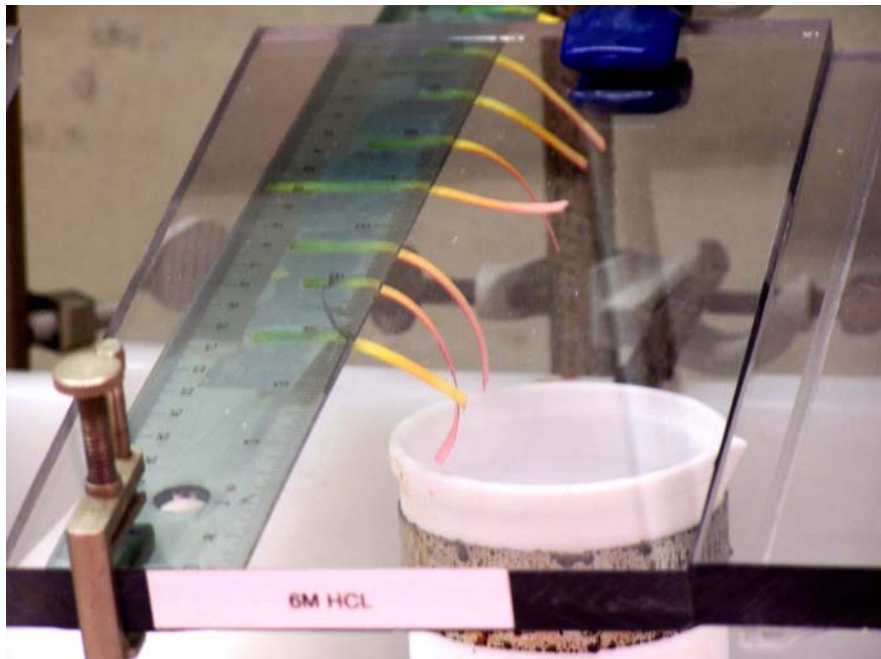


Figure 5-5. Series of Litmus Paper over the 6M HCl Vessels (24 hours after lowering the height to 2”)

After 28 days of exposure, there were no signs of visual degradation noted on any of the tear-offs or “blank” systems for any of the acids (see Figure 5-6 through Figure 5-9).¹⁵ These photos can be compared to Figure 4-2 which is the baseline condition. These photographs can also be compared to Figure 5-1 through Figure 5-4 (after 13 days of exposure at the 4” level).

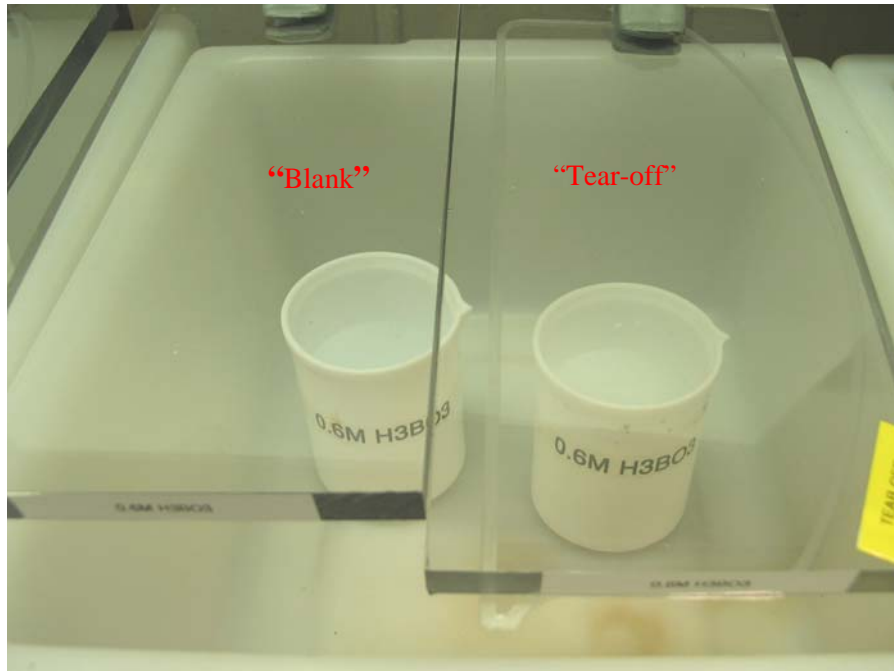


Figure 5-6. Visual Clarity of “Blank” (left) and “Tear-Off” (right) after 28 Days (0.6M H₃BO₃)

¹⁵ The 28 days of exposure are the combined duration at the 4” and 2” levels (see Table 5-1).

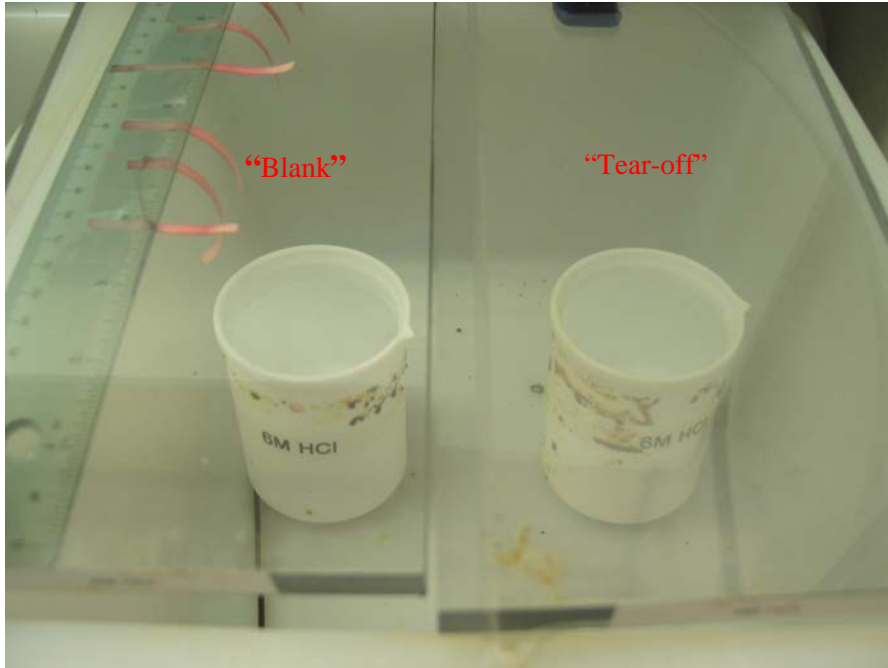


Figure 5-7. Visual Clarity of "Blank" (left) and "Tear-Off" (right) after 28 Days (6M HCl)

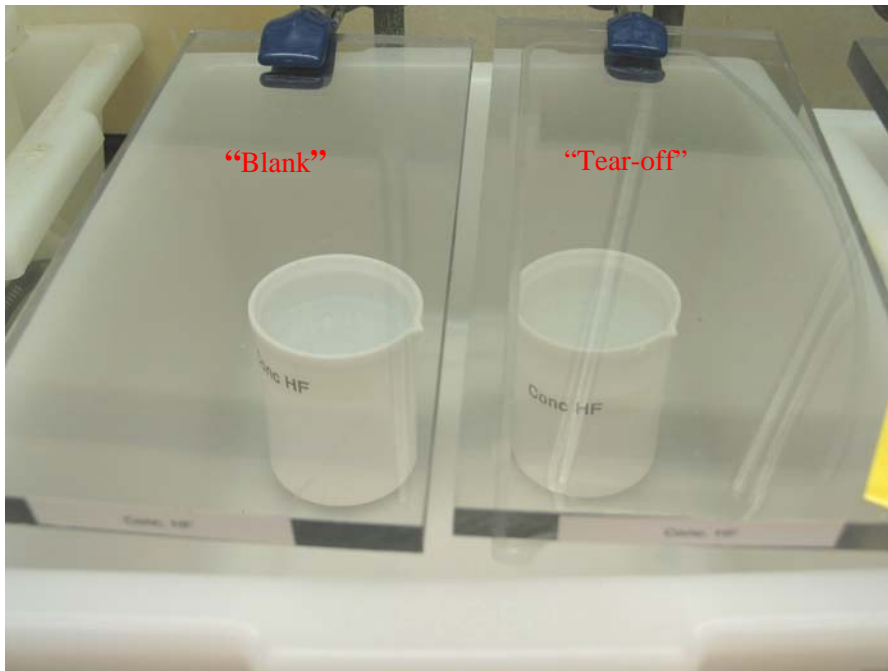


Figure 5-8. Visual Clarity of "Blank" (left) and "Tear-Off" (right) after 28 Days (Concentrated HF)



Figure 5-9. Visual Clarity of “Blank” (left) and “Tear-Off” (right) after 28 Days (Concentrated HNO₃)

After 39 days of exposure (at varying heights), a decision was to remove the outer layer tear-off in order to obtain an initial assessment of possible mechanical degradation as a result of acid vapor exposure. More specifically, could the outer layer tear-off be completely removed as a single sheet? Of particular interest (in terms of visual clarity) was the tear-off associated with the concentrated HNO₃ which had been exposed to an “accidental splash” as a result of a mechanical failure of the ring stand holding the acid vessel. The failure occurred on July 27, 2005 and resulted in visually noticeable droplets of HNO₃ adhering to the outer layer tear-off (see Figure 5-10). After 24 to 48 hours, the droplets had dried and no sign of hazing or distortion was readily apparent. There were no visual distortions when the tear-off was removed after 39 days of exposure (12 days after the splash). This result suggests that the tear-offs are relatively tolerant to “accidental” acid spills or splashes as measured by visual clarity. This observation is consistent with the technical information obtained on the Mylar tear-offs that “hazing” occurred only after complete immersion of Mylar in various acids – a situation that is unlikely to occur for the application being considered.

In terms of mechanical integrity, all outer layer tear-offs (including the HNO₃ splash) were easily removed as a single sheet (no tearing or ripping) after 39 days of exposure. It should be noted that given there were no visual distortion of the “blanks”, the test could not be classified as “overly aggressive” based on the short duration.

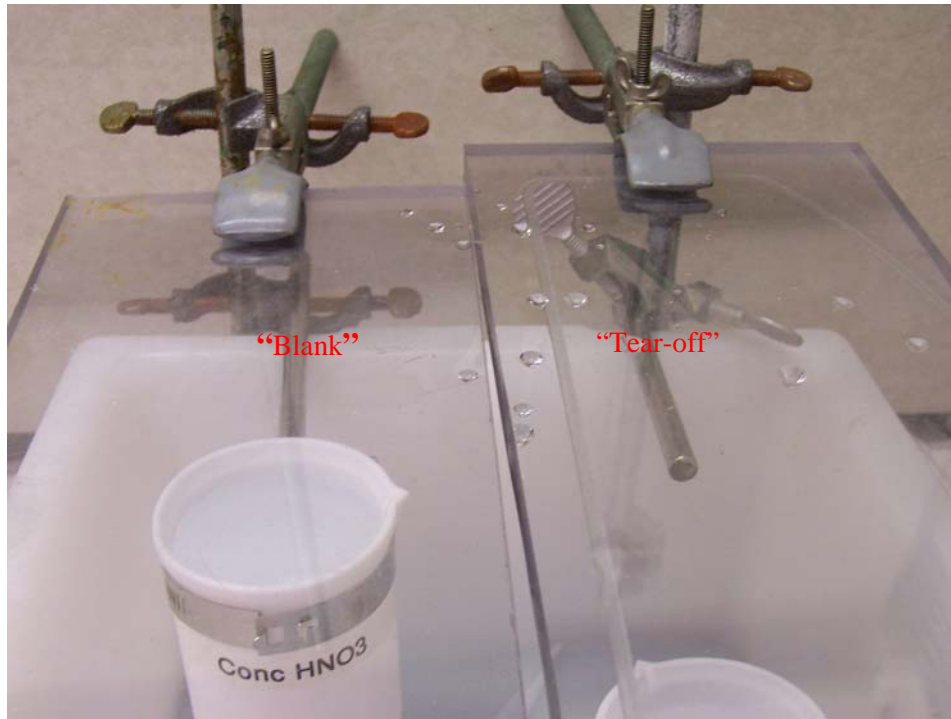


Figure 5-10. Concentrated HNO₃ Splatter on Tear-Off (July 27, 2005)

Based on the lack of any visual distortion of the tear-offs after 39 days of exposure, a decision was made to lower the systems from 2" to 0" (i.e., with each plate "resting" directly on the acid vessel with no physical gap or separation between the two). This scenario provides an extremely aggressive test condition (with the exception of an accidental spill or splash as noted above) to assess the potential impact of acid vapors.

After 17 days of exposure (at the 0" level), a circular "yellow stain" was very apparent on the "blank" but was not observed on the "tear-off" system. Figure 5-11 compares the "blank" (left hand side) and the "tear-off" system (right hand side). The stain results in a faint yellow circle within the circumference of the acid vessel. Also noted (for both systems) is condensation on the blank and tear-off.

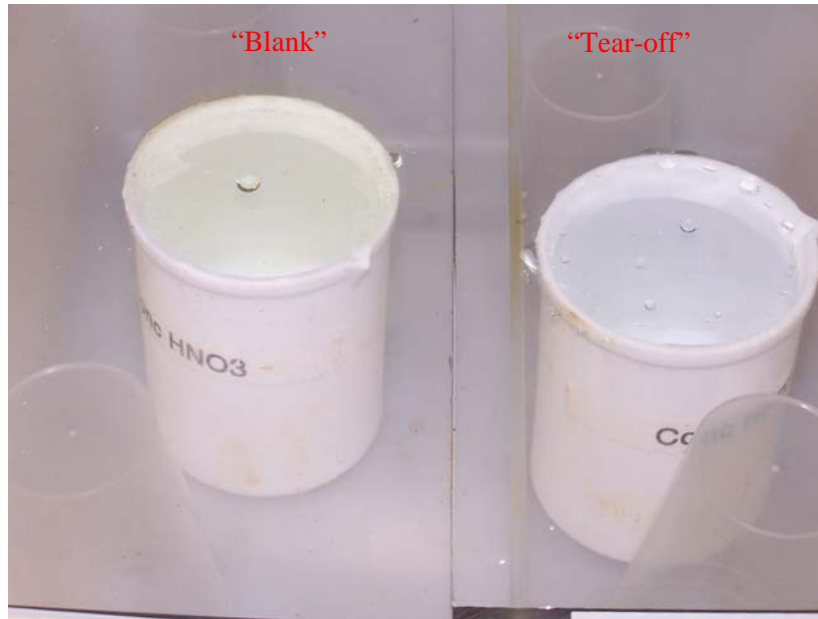


Figure 5-11. Circular “Yellow” Stain on the “Blank” after 17 Days (Concentrated HNO₃), 0” Mark

Figure 5-12 is another view of the concentrated HNO₃ “blank” system which shows the “yellow” stain and condensation. The stain appears to be the result of a reaction between the HNO₃ vapors and the Lexan “blank” – the color was not removed upon wiping with a Q-tip. Figure 5-13 is a photo of the “blank” after being lifted from the acid vessel. Note the large droplet of condensate (shown in Figure 5-11) is now absent, due to droplet running off the plate while the “blank” plate was removed from the test stand.

The discoloration of the “blank” after 17 days of exposure at the 0” mark suggest that this test condition may be overly aggressive based on historical observations in the SRNL shielded cells. Although aggressive, there does appear to be an advantage with the “tear-off” system, as this system remains “unchanged” based on visual observations (i.e., no yellow stain and no visual distortion noted). This suggests that the “tear-off” system may be more resistant to the HNO₃ vapor under these test conditions. It should be noted that the 0” height is not representative of actual service conditions, but may be approximate (or even bounding) for an acid “spill or splash”.

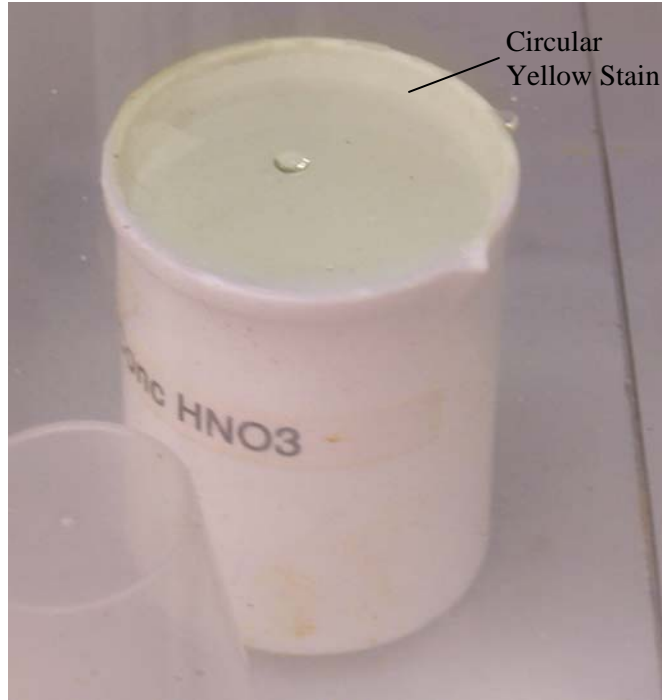


Figure 5-12. Circular “Yellow” Stain on the “Blank” after 17 Days at the 0” Mark (Concentrated HNO₃)

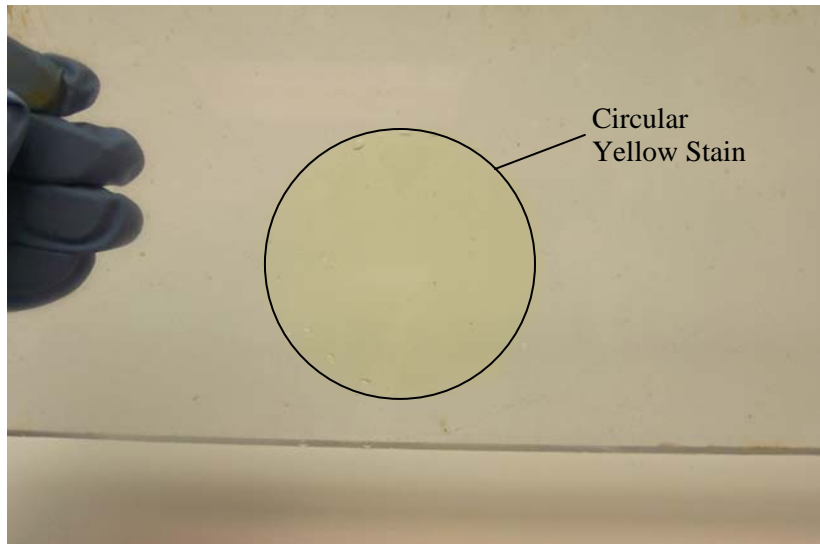


Figure 5-13. Circular “Yellow” Stain on the “Blank” after 17 Days at the 0” Mark (Concentrated HNO₃)

After 35 days of testing at the 0" mark,¹⁶ the only signs of visual degradation were observed in the "blank" for concentrated HNO_3 . The circular yellow pattern was still very apparent. All the other "blanks" and all "tear-offs" systems showed no signs of visual or mechanical degradation. Figure 5-14 through Figure 5-17 provides a measure of the visual clarity for the various systems after 35 days at the 0" height level.

The 35 days duration is relatively short when compared to the anticipated service life of Lexan sheet used in the shielded cells. However, the fact that there is no gap between the acid baths and the tear-off is a condition that is potentially unrealistic (and bounding) for typical service conditions – which may off-set or partially balance the short time periods to some degree. Regardless, the lack of hazing and/or physical degradation does suggest that the "tear-off" concept is feasible for these environmental or service conditions.



Figure 5-14. Visual Clarity of "Blank" (left) and "Lexan + Tear-Off" (right) after 35 Days at the 0" Mark (0.6M H_3BO_3)

¹⁶ The "2nd layer tear-off" was exposed on August 8, 2005 with the "final" visual descriptions for this report being documented on September 12, 2005 for a total of 35 days (at the 0" level). It is noted that there was no attempt at assessing mechanical integrity was made for the 2nd layer tear-off (now the outer layer) given visual degradation was not apparent. These tests will be monitored into FY06 to provide longer-term data.



Figure 5-15. Visual Clarity of “Blank” (left) and “Lexan + Tear-Off” (right) after 35 Days at the 0” Mark (6M HCl)



Figure 5-16. Visual Clarity of “Blank” (left) and “Lexan + Tear-Off” (right) after 35 Days at the 0” Mark (Concentrated HF)



Figure 5-17. Visual Clarity of “Blank” (left) and “Lexan + Tear-Off” (right) after 35 Days at the 0” Mark (Concentrated HNO₃)
(Note: Yellow circular stain on Lexan “blank”)

5.2 Base Resistance

After approximately 24 hours, the simulated sludge had dried and adhered to the “blank” and “tear-off” systems. Figure 5-18 shows the dried simulated sludge on the “blank” and on the “tear-off”. Deionized water and a wipe were used to wash the dried sludge off both systems. The result smeared the sludge across both surfaces leaving a residue which quickly dried. The outer layer tear-off was removed and the result is shown in Figure 5-19. The outer tear-off is shown on the right (note that the residual sludge is only on half of the tear-off due to its original placement – see Figure 4-3). Note that the outline of the remaining tear-offs (two layers) is very apparent.

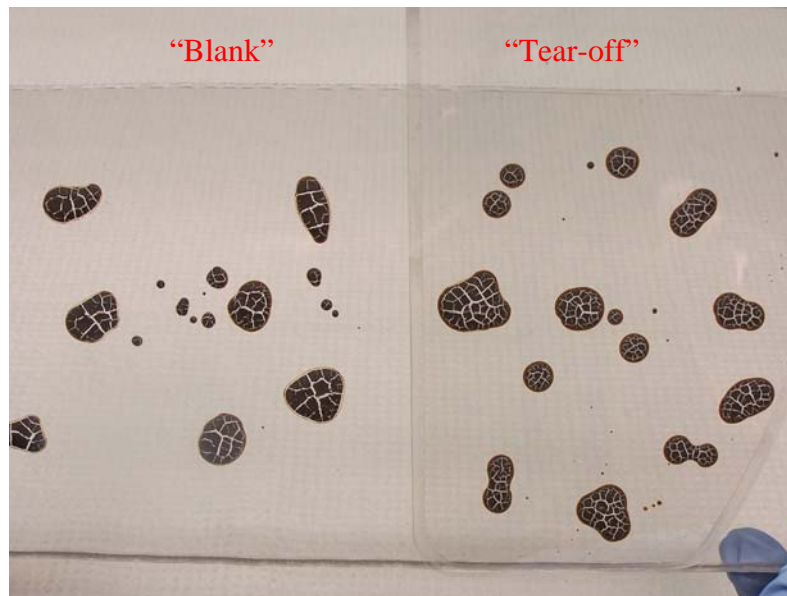


Figure 5-18. Dried Simulated Sludge on the “Blank” (left) and the “tear-off” (right)

Removal of the outer tear-off restored visual clarity to the upper portion as shown in Figure 5-19. The lower portion still has residual sludge covering the surface. An assessment of the tear-off was made to determine if the locations where the sludge was originally placed and dried could be identified. Given the adherence to the outer tear-off and “blank”, it was thought that “spots” may be detected – this was not the case. No visual signs of chemical or physical interaction were noted between the Mylar tear-off and the sludge. The two tear-offs remaining serve as the next layer of defense or protection for the subsequent event. In this case, the Lexan sheet (with the tear-off system applied) would not have to be removed and disposed of – a potentially significant advantage of the “tear-off” system concept.

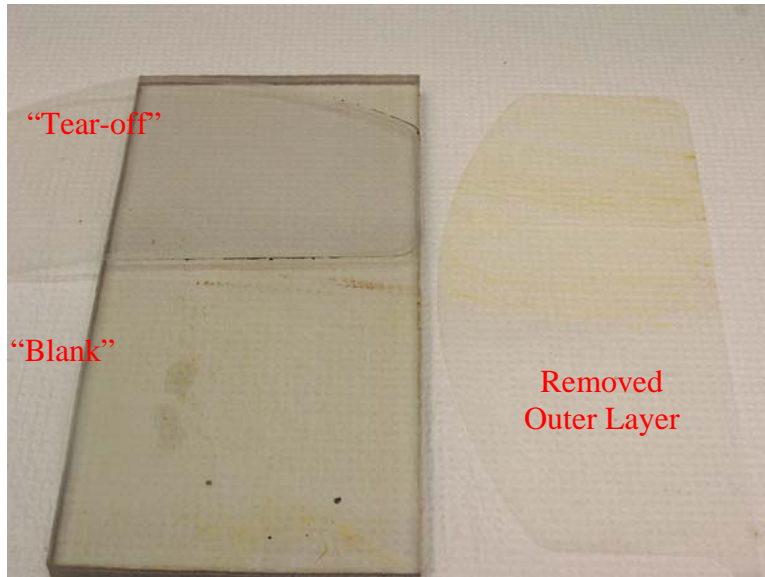


Figure 5-19. Restored Visual Clarity of the “Tear-Off” System (left, upper portion) Via Removal of the Outer Layer Tear-Off (right)

5.3 Scratch Resistance

As expected, the results of the “scratch resistance” testing confirmed that marks or scratches could be induced by the manipulators using “excessive” force on both the “blank” and “tear-off” systems. The scratches and marks observed on the “blank” were consistent with or at least similar to those observed in actual service based on SRNL shielded cell personnel observations. Figure 5-20 shows the results of the “intentional abuse” to the “blank” and “tear-off” systems. Scratches or marks are very apparent on both the “tear-off” and “blank”. As previously noted, the real measure of success in this test is the ability to completely remove the outer layer tear-off and as a result “erase” the marks and restore visual clarity. In addition, an assessment of the ability of the outer layer tear-off to minimize damage to the underlying layers and Lexan plate that could result in decreased visual clarity was made.

Figure 5-21 shows the same system after removal of the outer layer tear-off with the manipulators. The marks/scratches on the “tear-off” system have been removed refreshing visual clarity as conceptually intended (one measure is the reflection of the bottle on the upper portion of the system). In addition, there was no obvious damage to the underlying layers of the multi-layer tear-off system.

Although the tabs used to remove the outer layer tear-off were effective, they were not specifically designed for use with the manipulators. The shielded cells technician had no trouble grabbing the “improvised” tab and removing the outer layer tear-off in a uniform and peeling manner. If this system were to be implemented in the shielded cells environment, a more effective tab system should be designed into the manufacturing process. Removal of the outer tear-offs not only restored visual clarity without compromising mechanical integrity but also demonstrated the ability to remove the single outer layer sheet with the manipulator which preserves the multi-layer aspect of this concept.

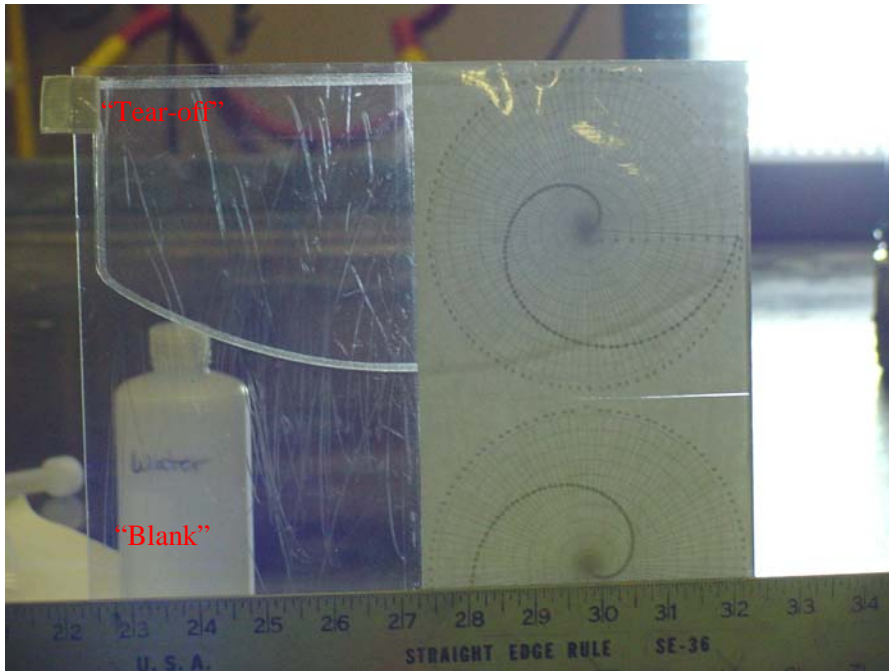


Figure 5-20. Lexan Blank and "Tear-Off" System After Manipulator Interaction

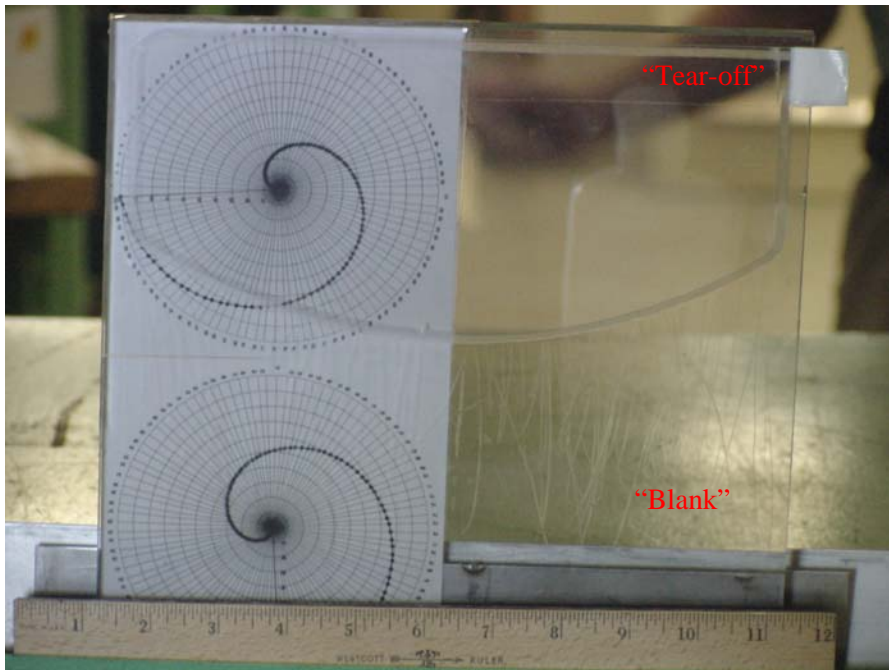


Figure 5-21. "Blank" and "Tear-off" Systems After Removal of Outer Layer

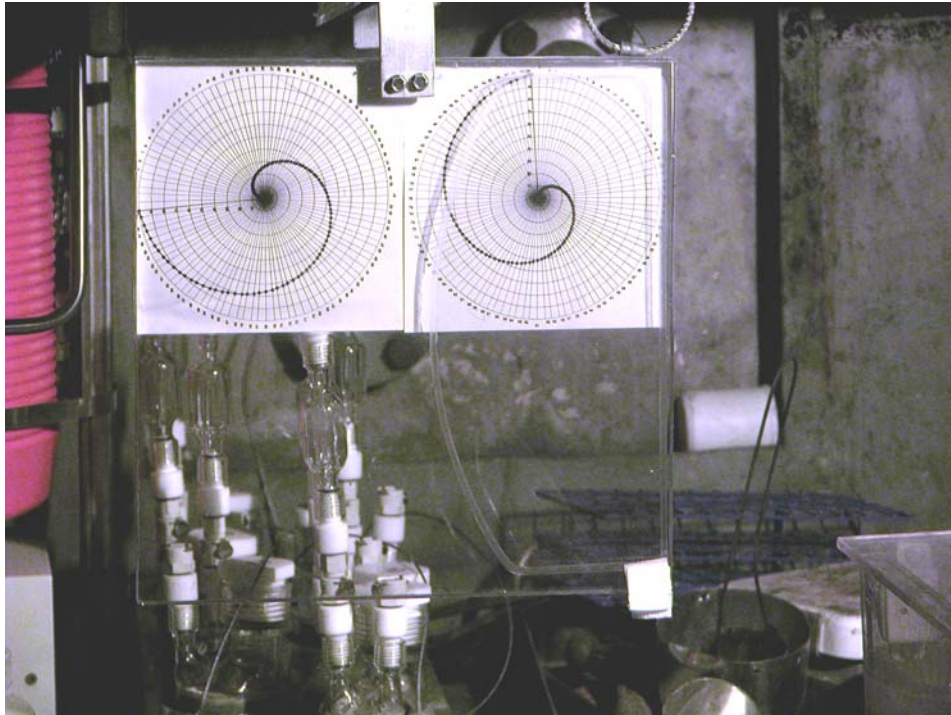
5.4 In-Situ Cell #15 Testing

Figure 5-22 shows the “blank” and “tear-off” systems hanging from an intermediate wall in Cell #15 just after cell entry (July 28, 2005). The “tear-off” is visible on the right hand side of the Lexan plate which is aligned vertically – see Figure 4-5 for a comparison photo. As previously mentioned, the average radiation dose in Cell #15 is 17.3 rad/hr (Bibler (2005)). The SRNL shielded cells personnel monitored the system on a daily basis for any signs of visual distortion or mechanical degradation.



Figure 5-22. “Blank” and “Tear-off” Systems in the SRNL Shielded Cells (Day 1)

After 46 days of exposure (estimated cumulative gamma dose of ~200,000 rad), there were no signs of visual distortion – see Figure 5-23 (photo taken prior to the removal of the outer layer tear-off on September 12, 2005). After documenting the visual clarity of the system, the shielded cell technician used the manipulators to remove the outer layer tear-off. The outer layer tear-off was completely removed (i.e., no ripping which demonstrated some degree of mechanical integrity after a cumulative dose of ~ 200,000 rad of gamma exposure) and left the two remaining tear-offs undamaged. The system remains in Cell #15 to support longer-term observations. Although the data is relatively short-term (46 days), there were no “show-stoppers” observed during the in-cell test after high gamma dose. For other applications (e.g., glovebox or radiochemical hoods), alpha contamination and/or beta radiation damage resistance may be of interest rather than the effects of gamma radiation as evaluated in the Phase 1 Cell #15 test.



**Figure 5-23. Lexan – Tear-off System in the SRNL Shielded Cells (Day 46)
– prior to removing outer layer tear-off**

6.0 SUMMARY

The Savannah River National Laboratory (SRNL) has conducted a series of scoping tests to assess the potential use of Mylar[®] tear-offs to serve as a primary or secondary protective barrier against acid etching (“frosting”), accidental scratching (e.g., by manipulator or tool contact), accidental spills/splashes (e.g., sludge and/or acids), and/or radiation/contamination damage (α and/or β) for shielded cells windows. The concept being investigated is to use thin, multi-layered sheets of Mylar (referred to as a “tear-off” system) which could be directly applied to the Lexan[®] sheet or glovebox/hood sash window to serve as a secondary (or primary) barrier. Upon degradation of visual clarity due to accidental scratching, spills/splatters, and/or radiation damage, the outer single sheet of Mylar could be removed “refreshing” or restoring the view. Although the concept of using a tear-off system as a protective barrier was conceptually enticing, potential technical issues were identified and addressed as part of this feasibility study. These included: (1) acid resistance, (2) base resistance, (3) scratch resistance, and (4) radiation damage resistance (via an in-cell demonstration). Not only can these four factors play a significant role in determining the visual clarity of the integrated system, they can also contribute to the mechanical integrity issues which could dictate the ability to remove the layers.

With respect to acid resistance testing, no visual or physical degradation was observed on the “tear-off” systems when exposed to concentrated HF, concentration HNO₃, 6M HCl, and 0.6M H₃BO₃. Although a relatively short-time period for exposure, the test conditions were considered “aggressive” and included an assessment of an accidental HNO₃ spill. The complete removal of the tear-offs after 39 days of exposure (at a 0” height) to all acids suggests that the mechanical integrity of the system was not degraded. The observation of a circular yellow stain on the Lexan “blank” under HNO₃ acid exposure at 0” after 17 days, clearly indicates that the Mylar tear-offs may be more chemically resistant than the Lexan sheet under those service conditions.

To assess the resistance of the tear-offs to highly basic materials, a simulated DWPF sludge with a starting pH of 12.9 was “splattered” onto the tear-off. The sludge was partially removed through the use of deionized water and a wipe leaving a smear of sludge across both the tear-off and Lexan “blank”. Upon removal of the outer tear-off, visual clarity was restored on the “tear-off” system leaving the sludge residue on the Lexan “blank”. Upon visual inspection of the tear-off, no visual sign of reaction or degradation could be detected. The results indicate that the Mylar tear-off was impervious to the high pH sample.

The “scratch resistance” testing showed that marks or scratches can be induced by the manipulators (in “mock-up”) on both the “blank” and “tear-off” systems. The scratch marks were consistent with those observed in actual service in the SRNL shielded cells. Upon removing the outer tear-off layer with the manipulators, visual clarity was restored. The results of this test not only confirmed the conceptual “erasing” of marks or scratches but also demonstrated the ability of the manipulators to grasp and remove the outer layer tear-off.

Although a short-term duration, the results of the in-situ Cell #15 tests suggest that the concept of the tear-off is feasible under realistic service conditions as measured by high gamma radiation background. The tear-offs showed no signs of visual degradation over the 46 day test period (with a cumulative gamma dose estimated to be ~ 200,000 rad). In addition, the outer layer tear-off was successfully and completely removed suggesting no mechanical degradation after the gamma radiation exposure. The ability to remove the outer layer tear-off (using an “improvised” tab) supported the observations during the “mock-up” scratch resistance testing that the manipulators can effectively remove the outer layer. It should be noted that the tear-offs do not provide a protective barrier to the gamma radiation as the

penetration depths are too great. The outer layer tear-off would provide a protective barrier against α and/or β radiation (conditions recommended for further testing –see Section 7.0).

The results of the Phase 1 study clearly indicate that the Mylar tear-off concept is a potential technical solution to mitigate excessive damage that would result from acid etching or spills, base damage (as a result of a sludge spill or splatter), gamma radiation damage, and/or accidental scratching (due to manipulator/tool contact). The short term tests performed in this task showed that Mylar tear-offs can withstand the chemical and physical abuses expected in off-normal shielded cells operations. The “tear-offs” not only provide some measure of acid resistance, as reflected by the lack of visual degradation after being exposed to four acids, but also act as a protective barrier to accidental contact with the manipulators and/or tools. The conceptual “erasing” of scratches or marks was demonstrated in the shielded cell mock-up facility through the removal of the outer layer tear-off with manipulators.

The successful removal of the outer layer tear-off with the manipulator, using tabs not specifically designed for such a purpose, demonstrates that the system is “manipulator-friendly” and could be implemented in the shielded cells. The ability to remove the outer layer tear-off not only regains visual clarity but also reduces waste disposal volumes (i.e., disposal of a thin sheet of Mylar which is “collapsible” versus the bulk disposal of a rigid Lexan sheet or glovebox/sash window) which is more cost effective. The “tear-off” system could also reduce the number of cell entries needed to replace the Lexan sheet and increase the time interval between glovebox/sash window change outs which can be costly and time consuming.

7.0 RECOMMENDATIONS

The following recommendations are made to advance the potential use of the Mylar tear-offs as a primary or secondary protective barrier. Prior to implementation, the following items should be considered:

- Phase 1 results were reviewed with DWPF. Based on that discussion, additional tests or environmental conditions were identified for consideration during the Phase 2 scope. These included:
 - resistance to formic acid, NaOH, sodium peroxide
 - provide DWPF with two Lexan plates with tear-offs system mounted on half of the plate for in-situ testing in the DWPF analytical cells.
- Continue to monitor the acid resistance testing at the 0" height long-term. These tests are currently on-going and take very little effort to monitor.
- Obtain additional data on gamma radiation effects using Co-60 source and potential interactive effects (such as radiation damage coupled with acid exposure) on visual clarity and mechanical integrity.
- Obtain data on alpha and/or beta radiation damage/contamination to the tear-offs. This may be of particular interest for glovebox usage (e.g., actinide bearing materials).
- Continue to monitor the "in-situ" Cell #15 demonstration.
- Identify potential "End Users" (both commercial and government based) and potential companies/partners that would design and fabricate the Mylar sheets for specific applications (e.g., DuPont Teijin Films). Included in the design would be the size, mil thickness, number of tear-offs, modifications to the tabs, and compositional changes (if warranted).
- Identify companies that are potential licensees for the product.
- Perform assessments on thermal stability / fire resistance in case service conditions call for elevated temperatures. DuPont states that the melting point of their Mylar film is approximately 250°C. If service conditions approach or exceed this value, then the application of Mylar tear-offs may be limited.

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9.0 REFERENCES

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