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# F-Canyon 8.5 Evaporator Vessel Repair Patch Recommendation (U)

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#### **1 EXECUTIVE SUMMARY**

A leak was discovered in the 8.5 evaporator pot on the warm side of F-Canyon during a caustic flush procedure intended to dissolve solids that had deposited during routine operation. A repair patch was developed based upon the expected operational parameters of the 8.5 evaporator. The recommended repair patch is shown in Figure 1. The repair patch consists of a stainless steel backing plate with a  $\frac{1}{2}$ " Viton<sup>®</sup> B Foam, followed by Pelseal 2012 Viton<sup>®</sup> sealant, and a centered plug of RTV sealant. The foam has  $\frac{1}{4}$ " –  $\frac{1}{2}$ " thick layer of Pelseal 2012 sealant troweled onto the foam surface. The material will be applied with a bevel, i.e. the material will be thicker towards the top since the sealant creeps over time when held in a vertical position. However,  $\frac{1}{4}$ " of foam area is left uncovered along the perimeter of the foam surface. The recommendation for materials to be used in the repair patch is based upon limited immersion testing and ongoing lead surveillance testing.

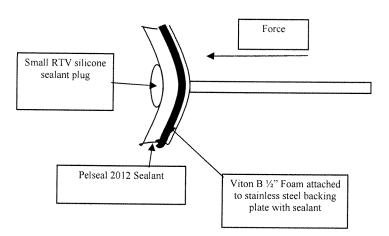


Figure 1: Recommended Repair Patch and Materials

#### 2 INTRODUCTION

A leak was discovered in the 8.5 evaporator pot on the warm side of F-Canyon during a caustic flush procedure intended to dissolve solids that had deposited during routine operation. A program was developed to determine possible repair options for the leaksite. The program included immersion and lead surveillance testing with candidate repair materials in simulated evaporator conditions. In addition, a remote delivery apparatus was designed and fabricated to implement the chosen repair patch. The experimental results, recommendations for repair patch materials, and the associated challenges are discussed herein. The delivery system is discussed in a separate report.

#### 2.1 Evaporator Pot Materials of Construction

The evaporator pot is made of 3/8-inch type 304L stainless steel (SS) hot rolled plate welded with type 308-SS filler rod. The specification of the material is made to original DuPont specification SW 820M, that was replaced with SW 301M "Selection Criteria for Type 300 Series Stainless Steels," and SW 800M, "Corrosion Evaluation of Stainless Steels and Nickel Based Alloys".<sup>1</sup> The type 304L-SS, a low-carbon modification of type 304-SS, was used to avoid sensitization during welding. The general corrosion resistance is similar to type 304-SS, but no post-weld heat treatment is required for type 304L-SS prior to service. The type 304-SS family has historically provided good corrosion resistance when exposed to nitric acid in environments associated with nuclear material processing. Table 1 provides the nominal chemical compositions of the primary constituents of types 304-SS and 304L-SS and typical mechanical properties.

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SS type	Cr (wt %)	Ni (wt%)	C (max)	Other	σ <sub>UTS</sub> (ksi)	σ <sub>y</sub> (ksi)	Elong. (%)
304	18-20	8-10.5	0.08	Mn = 2	85	35	55
				Si = 1			
				P = 0.045			
				S = 0.03			
304L	18-20	8-10.5	0.03	Mn = 2	85	35	55
				Si = 1			
				P = 0.045			
				S = 0.03			

Table 1: Chemical Composition	n and Typical Mechanical Pr	operties for types 304/304L-SS <sup>1</sup>
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#### 2.2 Visual Observation of Leaksite<sup>2</sup>

The initial view of the leak appeared to show two small holes (shown in Figure 2a) in the shell of the evaporator. The perforations are estimated to be no larger than 3/32" in diameter and no more than 1/4" apart, located approximately 1-1/2" above, and oriented parallel to the bottom head-to-shell weld. This weld is estimated to be 3/4" in width and still has a weld crown though the weld ripples and any undercut were obviously removed with a grinder during fabrication. The perforations are located at the boundary or slightly beyond the heat affected zone (HAZ) of the bottom head-to-shell weld. The design drawing indicates several internal attachments welded on the interior of the shell in this immediate area. Such internal attachments are typically made using oversized fillet welds that can create an additional HAZ in the shell that are not readily discernable from the exterior. It is suspected that the holes may well be in just such a HAZ, which is more susceptible to corrosion.

After additional cleaning with a power wire brush, the area between the holes was opened up to reveal a single crescent shaped perforation (shown in Figure 2b) approximately 1/4" in length. Bare metal was attained all around the hole without any residual deposits. It is unclear whether this change was caused by removing a very thin layer of material between the initial holes or by removal of deposits. Additional cleaning was again performed and while the crescent shaped perforation did not appear to increase in size, it did begin to show some "denting" or displacement of the area immediately surrounding the hole. This change indicated that the shell wall in this localized area is thinned.

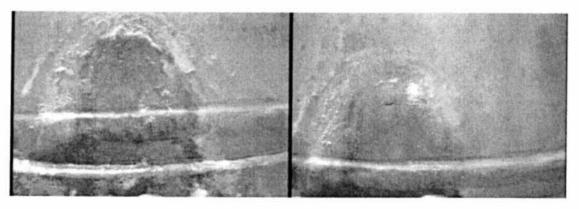


Figure 2: Leaksite Before (2a) and After Cleaning (2b)

#### 2.3 Corrosion Evaluation of Leaksite

The current 8.5 evaporator vessel has been used in concentrated nitric acid service for 20 years. Canyon evaporators are limited to a maximum of 35% HNO<sub>3</sub>, based on corrosion data indicating that severe corrosion of type 304L SS

occurs at concentrations of 40-50% HNO<sub>3</sub>. The operational parameters are based upon coupon testing performed on type 304L SS in the annealed, as-welded, and sensitized conditions in simulated solutions.<sup>3</sup> The testing concluded the following:

- 1. Corrosion rates increased rapidly when nitric acid concentrations exceeded 45%.
- 2. Corrosion rates generally increased with increasing nitric acid concentration.
- 3. Severe intergranular attack of the sensitized coupons was noted.

This portion of the evaporator pot is expected to have the microstructure most susceptible to corrosion due to the numerous weldments on the interior of the tank wall with the attachment of the overflow weir. A through-wall penetration of the 3/8" thick wall could develop in approximately 19 years under the following conservative assumptions: (1) corrosion rate of 60 mils/year, as observed in sensitized type 304L coupons in simulated solution, (2) 33% HNO<sub>3</sub> exposure at boiling temperatures, and (3) uniform exposure at this region. It is possible that the extensive weldments in this area could lead to a buildup of impurities, leading to a high corrosion rate. However, it is expected that the remainder of the tank will be microstructurally less susceptible to corrosion.

#### **3 REPAIR METHOD DEVELOPMENT**

The strategy for developing an effective repair patch for the 8.5 evaporator is shown in Figure 3. The materials selection process was based upon the initial functional requirements. Immersion and lead surveillance testing was performed in line with the functional requirements to determine the most effective repair patch solution.

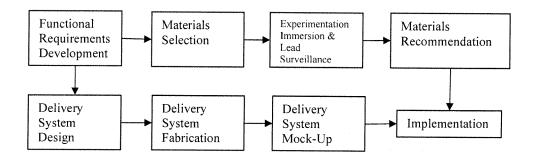


Figure 3: Strategy for Development of Repair Method

#### 3.1 Functional Requirements for Repair Configuration and Materials

The following are the typical operational conditions of the 8.5E that a patch material must withstand for a duration of 3 months.<sup>4</sup> A temporary repair is desired for at least 3 months of operation to meet scheduled waste reduction.

- Temperature: The evaporator operates in a range of 100-111°C with procedural temperature limits of 112–
  120°C. When the evaporator is heated, steam is applied and it is brought up to boiling in 30-60 minutes.
  When the evaporator is shutdown, normally the steam is valved "off" and the evaporator cools without any
  forced cooling (cooling water). Alternatively, when it cools down to 80 degrees C, cooling water is
  supplied to the steam chest/tube bundle to increase the cooling rate.
- 2. *Pressure:* The pressure in the free board between the bubble cap trays and the liquid is typically less than 1 psig ranging from 6" to 27" water column. The liquid depth in the evaporator is 97" (bottom of the evaporator to overflow at the weir) with recent operations at 15-20 inches above the weir.
- 3. *Density:* The feed to the evaporator has a density of approximately 1 (similar to water or very weak acid). The solution is concentrated to a density ranging from 1.25-1.30.
- 4. *Composition of Solution:* The normal solution in the evaporator is 3-6M nitric acid with 0-10 g Fe/liter, 0-10 g U/liter, NaNO<sub>3</sub> salt, with some n-paraffin and tri-butyl phosphate (TBP) organics (usually only that

which is soluble in the aqueous feed). The organics tend to float on the surface due to density variation. Therefore, organic exposure is thought to be minimal. The flush/decontamination solutions is a minimum of 15 wt. % NaOH heated to boiling.

- 5. *Radiation:* There is alpha activity of approximately 1E+6 d/m/ml (primarily Am-241, Pu-238, -239, -240), and gamma activity around 1E6 1E7 d/m/ml. This translates to a field of approximately 5 R/hr.
- 6. *Remote Deliverability:* There is limited access and use of tools around the evaporator pot.

The 8.5 evaporator is typically run 2-3 times a week. However, with the extended downtime, it will be likely run continuously after initial startup. In addition, the caustic flush operation intended to remove deposits will be completed initially. The deposits were found to be phosphorus containing organic material, i.e. tri-butyl phosphate (TBP) and n-paraffin. A caustic flush could be used to hydrolyze both organic types without generation of NO<sub>2</sub> fumes.<sup>5</sup>

#### 3.2 Repair Concept

Based upon the functional requirements, several repair concepts were developed. Of these concepts the final simplified concept of the proposed repair option (as viewed from above) is shown in Figure 4.

The basic concept uses a multilayered material system with materials that are resistance to degradation in typical operational conditions. The material system must be sufficiently pliable be forced into the leaksite with horizontal force.

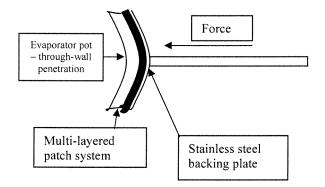


Figure 4: 8.5 Evaporator Vessel Repair Concept

#### 3.3 Materials Selection

Repair patch options based on commercially available polymeric materials were given primary consideration for simplicity and remote deliverability. Compliant or elastomeric materials were favored to accommodate surface imperfections and irregular geometries.

The initial down-select was focused on materials resistant to nitric acid service. Nitric acid is highly oxidizing and aggressive toward most polymeric materials, particularly at elevated temperatures. Resistance to caustic at elevated temperature for at least brief periods of time was also highly desired. Polymers that exhibit resistance to both strong nitric acid and strong caustic solutions are typically not available in forms deliverable for this application.

The most chemically resistant polymers for strong nitric acid or even dilute solutions at elevated temperature are generally fluoropolymers such as Teflon<sup>®</sup>. Teflon<sup>®</sup> PTFE is the most chemically-resistant polymer and would easily handle the chemistry involved in the tank repair. Other Teflon<sup>®</sup> types (FEP, PFA) are also highly resistant to nitric acid and caustic solutions, within service temperature limits. However, Teflon<sup>®</sup> is susceptible to creep and exhibits

limited resistance to radiation. Teflon<sup>®</sup>-impregnated asbestos gaskets are successfully used throughout canyon processes, but are protected from radiation degradation by their composite structure. They are also relatively stiff and thus have limited compressibility. Expanded PTFE (Gore-Tex<sup>®</sup>-type products) are available and much more compressible, but still limited in radiation resistance. Other fluoropolymers with high resistance to nitric/caustic solutions such as ETFE (Tefzel<sup>®</sup>), PVDF (Kynar<sup>®</sup>), and ECTFE (Halar<sup>®</sup>) also have superior radiation resistance to that of Teflon<sup>®</sup>, but are much stiffer and less compressible. PVDF materials also exhibit stress-cracking in strong NaOH solutions.

Elastomeric materials with resistance to nitric/caustic solutions were given primary consideration due to their compressibility. Only a few of the generic elastomers are considered resistant to strong nitric acid solutions, with even fewer having good resistance at elevated temperatures. These include fluoroelastomer types such as FKM (Viton<sup>®</sup>-type), FFKM (Kalrez<sup>®</sup> perfluoroelastomers), TFE/P (Aflas<sup>®</sup>-type), and other halogenated polymers such as Hypalon<sup>®</sup> (chlorosulfonated polyethylene) and Koroseal<sup>®</sup> (plasticized polyvinyl chloride). Of these, the fluoroelastomers are more resistant, particularly at stronger concentrations and higher temperatures. Butyl rubber also has excellent resistance to strong nitric acid, but is limited to service temperatures less than 120°F and has limited radiation resistance. Other elastomers such as EPDM (ethylene-propylene diene monomer) have excellent resistance to caustic solutions, even superior to that of the fluoroelastomers in some cases, but have limited resistance to nitric acid.

Only a few of the materials considered resistant to both nitric acid and caustic solutions are available as sealants, caulks or putties for leak sealing applications. The fluoroelastomers in particular (such as Viton<sup>®</sup>) are difficult to process in liquid form and can only be dissolved by very aggressive solvents such as MEK or methylene chloride. The fluoroelastomers are also difficult to process in lower hardness grades of solid material, and are typically only available in Durometer hardness grades of 60-80A. While such grades are commonly used for sealing applications such as O-rings and gaskets, they were judged to be too stiff for this application.

Additional materials such as flexible graphite (Grafoil<sup>®</sup>-type) and ceramic-based adhesives were also considered. Each has limitations of either chemical resistance, immersion/water resistance, or compliance. Materials commonly used for repair of machinery and vessels such as epoxies (fiber, metal, or ceramic-filled versions) were also considered, but exhibit limited resistance to nitric acid. Novolac-based epoxies and vinylester resins are more resistant to nitric acid than conventional epoxies, but are still generally less resistant than fluoropolymers and fluoroelastomers.

The materials considered (after downselect from the initial search) for the repair patch were (1) Viton<sup>®</sup> sealant, (2) Viton<sup>®</sup> A and Viton<sup>®</sup> B foam elastomer, (3) liquid metal paste consisting of stainless steel powder within a novolac epoxy binder, (4) silicone RTV sealant. Limited immersion testing and lead surveillance testing was done to determine the effectiveness of each of these materials as leak-seals.

#### 3.3.1 Viton Sealant

The Viton<sup>®</sup> sealant, Pelseal 2012, is a liquid fluoroelastomer sealant (Viton<sup>®</sup> A) that is combined with an accelerator to cure into a cross-linked polymer. Viton<sup>®</sup> is known to be resistant to nitric acid solutions, but possibly subject to degradation in caustic environments. Typically, the degradation will occur by softening, and possible loss of adhesion. The material can be delivered by trowel, and is viscous enough to be applied to a vertical surface. The following are the important parameters as published by the vendor:

<b>Properties</b>	<u>Value</u>
Viscosity	>2,000,000 cps
Weight/volume non-volatile matter	65%/45%
Tensile Strength	1200 psi
Elongation	50%
Specific Gravity	1.28
Operating Temperature	-40-206°C

The parts are mixed in the ratio of 27 parts by weight of base materials to 1 part by weight of Accelerator #4, i.e. one quart base material to 46 g (59 ml) of accelerator. The useful pot life of the mixed material is 4 hours at room temperature. A clean, dry surface is ideal for maximum adhesion, and sandblasting/degreasing is the minimum recommended surface preparation. The complete recommended cure time is 72 hours at room temperature.

#### 3.3.2 Viton A/B Sponge Elastomer

Viton<sup>®</sup> is a Dow-Corning product that refers to a series of fluoroelastomers based on the copolymer of vinylidene fluoride and hexafluoropropylene with the repeating structure of -CF2-CH2-CF2-CF(CF3)-. Historically, fluoroelastomer compounds (such as Viton) have only been available as solid extrusions, and as solid or sponge molded parts. However continuous sponge fluoroelastomer extrusions for closed cell foams have become recently available. As with other sponge seals and gaskets, these fluoroelastomer sponge extrusions require less closing force than do their solid counterparts to deliver an effective seal.

Viton  $B^{\text{(B)}}$  is also a terpolymer of vinylidene fluoride, hexafluoropropylene and tetrafluoroethylene monomers, offering broader chemical resistance than Viton<sup>(B)</sup> A types and has slightly higher resistance to strong nitric acid solutions. Lead oxide cured compounds also tend to exhibit better resistance than MgO cured products.

#### 3.3.3 Liquid Metal Paste

The liquid metal paste under consideration is Cotronics Epox-Eez RK456<sup>®</sup>, a stainless steel based epoxy repair putty. The epoxy binder is a novolac type binder that is more chemically resistant than the typical bisphenol type epoxy binder. The putty cures at room temperature to a highly machinable stainless steel based composite that the vendor claims to be usable to 500°F.

#### 3.3.4 RTV Silicone

Silicone rubber is a semi-organic synthetic. Its structure consists of a chain of silicone and oxygen atoms rather than carbon and hydrogen atoms, as in the case with other types of rubber. The molecular structure of silicone rubber results in a very flexible, but weak, chain. Silicones are very stable at low and high temperatures. Although fillers may improve properties somewhat, tear and tensile strengths remain relatively low. Silicones are not generally recommended for strong nitric acid, but are generally useful for caustic solutions. Therefore, this materials was considered primarily for the initial caustic flush exposure to provide additional protection to the Viton<sup>®</sup> material.

#### 4 MATERIALS TESTING

Immersion and radiation testing was done on the candidate materials to determine degradation of the materials when exposed to the relevant environments. In addition, lead surveillance testing on simulated leaksites was performed, and will continue to run, to determine functionality of the candidate patch combinations. An abridged test matrix was developed to ensure technically defensible quick results.

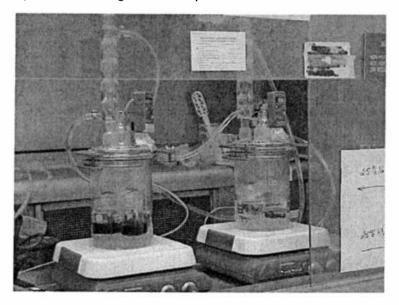
#### 4.1 Immersion Testing

Immersion testing was performed to determine the chemical and radiation resistance of the materials for the application conditions. The surface area exposed to the environment when applied to the evaporator will be limited, and full immersion testing resulted in a conservative approximation of degradation. The immersion testing consisted of exposure of small-size samples of candidate materials to 25% NaOH and 25% HNO<sub>3</sub> solutions, at near boiling conditions. Dimensional changes, weight change, and visual appearance were monitored. A summary of the immersion testing is shown in Table 2.

Exposures	Temperature	Time
25% NaOH	100°C	48 hours
25%HNO <sub>3</sub>	100°C	48 hours
Gamma Radiation + 25% NaOH (Viton <sup>®</sup> only)	RT	1E6/1E7 rads
Gamma Radiation + 25% HNO <sub>3</sub> (Viton <sup>®</sup> only)	RT	1E6/1E7 rads

# Table 2: Summary of Immersion Test Conditions

The immersion test setups are shown in Figure 5. The exposures were maintained at 100°C for 48 hours



### Figure 5: Exposure Test Setups

The results of the immersion testing are shown in Table 3.

Table 3:	Exposure	Test	Results	(all	weights	in grams	3)

Material	Initial Welght	Exposures	Final Welght	Weight Change	Material Characteristics	Solution Characteristics
*Pelseal Vendor #1	14.98	25% NaOH	20.12	5.14	Hard but flexible, cracking on sides	Blackened
*Pelseal Vendor #2	14.33	25% HNO3	18.16	3.83	Absorbed solution, flexible	Yellowed
Pelseal SRTC #1	1.53	25% NaOH	2.32	0.79	Hard, slightly embrittled	Blackened
Pelseal SRTC #2	1.68	25% HNO₃	2.32	0.64	Hard but flexible	Yellowed
RTV Silicone #1	0.86	25% HNO3	.65	-0.21	Softened, cracked	Yellowed
RTV Silicone #2	0.77	25% HNO3	.53	-0.24	Softened, cracked	Yellowed
RTV Silicone #3	0.87	25% NaOH	.84	-0.03	Slight Softening	Yellowed

Material	Initial Weight	Exposures	Final Weight	Weight Change	Material Characteristics	Solution Characteristics
RTV Silicone #4	0.9	25% NaOH	.86	-0.04	Slight Softening	Yellowed
Liquid Metal Paste	2.69	25% HNO <sub>3</sub>	N/A	N/A	Epoxy Binder Dissolved	Residue in bottom
Liquid Metal Paste	2.84	25% HNO <sub>3</sub>	N/A	N/A	Epoxy Binder Dissolved	Residue in bottom
Liquid Metal Paste	2.9	25% NaOH	2.94	0.04	Surface attack only	Greenish
Liquid Metal Paste	2.89	25% NaOH	2.89	0.01	Surface attack only	Greenish
Sponge Viton <sup>®</sup> A #1	0.95	25% NaOH	1.41	0.46	Crumbling/Layering	Blackened
Sponge Viton <sup>®</sup> A #2	0.86	25% NaOH	1.19	0.33	Crumbling/Layering	Blackened
Sponge Viton <sup>®</sup> A #3	1.09	25% HNO3	N/A	N/A	Absorbed solution & swelling	Yellowed
Sponge Viton <sup>®</sup> A #4	1.14	25% HNO₃	N/A	N/A	Absorbed solution & swelling	Yellowed
Sponge Viton <sup>®</sup> B #1	1.63	25% HNO3	2.88**	1.25	Absorbed solution & swelling	Blackened
Sponge Viton <sup>®</sup> B <b>#2</b>	1.68	25% HNO3	3.14**	1.46	Absorbed solution & swelling	Blackened
Sponge Viton <sup>®</sup> B #3	1.62	25% NaOH	1.71**	0.09	Salt crystals on surface, crumbling	Yellowed
Sponge Viton <sup>®</sup> B #4	1.65	25% NaOH	2.95**	1.3	Salt crystals on surface, crumbling	Yellowed

\* Vendor cured test samples

\*\* Weights measured after 120 hour air dry.

The blackening of the solutions of NaOH with Viton<sup>®</sup> is typically due to the leeching of carbon black in addition to the vinylidene fluoride and possibly curing oxides.

## 4.2 Radiation Testing of Pelseal Viton<sup>®</sup> 2012 Sealant

Radiation exposures were done only on the Viton<sup>®</sup> materials, because epoxies are typically highly resistant to degradation when exposed to radiation. In addition, the Viton<sup>®</sup> materials were considered the primary candidate for a viable repair patch after the materials down-select. Exposures were done in 25% HNO<sub>3</sub> and 25% NaOH, and in a Co-60 gamma irradiation source to a total dose of 1E6 and 1E7 Rads, at a dose rate of 7.44E5 Rads/hr.

Dynamic mechanical analysis (DMA) was used to determine thermo-mechanical properties of the Viton<sup>®</sup> sealant material before and after exposure. The DMA provided information on the ability of materials to store and dissipate mechanical energy upon cyclic deformation. The storage modulus, loss modulus, and tan delta were measured as a function of temperature, and a glass transition temperature was determined. The storage modulus is defined to be the energy stored during a cycle, and the loss modulus is the energy lost. The quantity tan delta is the ratio of the loss modulus to the storage modulus and indicates the ability of the polymer to dissipate energy as heat. The results of the testing are shown in Table 4: Some of these results may also be a function of the degree of cure rather than exposure.

Exposure	Glass Transition Temperature (°C)				
Unexposed #1	-31				
Unexposed #2	-30				
NaOH,1E7 Rads, #1	-34				
NaOH, IE7 Rads, #2	-32				
HNO3, 1E7 Rads, #1	-31				
HNO <sub>3</sub> , 1E7 Rads, #2	-26				
NaOH, 2 days 100°C, #1	-27				
NaOH, 2 days 100°C,#2	Two transitions, not readable				
HNO <sub>3</sub> , 2 days 100°C, #1	Two transitions at -51 and -14				

Table 4: Glass Transition Temperature of Pelseal Viton<sup>®</sup> Before and After Exposure

The glass transition temperature indicates a change in the polymer properties into a rubbery state from a hard state. It is seen that the Viton<sup>®</sup> is rubbery at room temperature. Any change in crosslinking properties would be exhibited as a change in the glass transition temperature. The results indicate that exposure to HNO<sub>3</sub> and NaOH at room temperature and 1E7 rads of gamma irradiation do not have an effect on the glass transition temperature. However, high temperature immersion exposure had some effect on the crosslink properties. The twin transition curve may be indicative of a change in the crosslinking properties or of moisture in the samples.

#### 4.3 Lead Surveillance Testing

Lead surveillance testing was performed to determine functionality of several combinations of materials and exposures. The testing was performed by machining a through-wall notch into a 1L stainless steel beaker and applying various repair patch combinations (as shown in Figure 6) in 'Cells 1-4' respectively. Each cell has 2 machined leaksites (A and B) that have been patched.

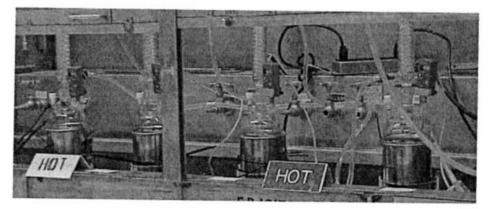


Figure 6: Exposure testing of candidate materials

The exposures and material combinations are shown in Table 5. Thermal cycling was simulated by allowing exposure to heated solution during the day and allowing a slow cool overnight by turning off the temperature daily. The solutions used were 25 % NaOH and 25% HNO<sub>3</sub>. Each of the patch combinations was applied with a stainless steel backing plate and held in place with a cable tie with minimal horizontal force. The sealants were fully cured according to recommended vendor specifications. The seals were monitored for possible leakage and any changes in appearance of solution or of the patch material. All patch materials seem intact for 2 weeks with no observed leakages. The lead surveillance tests continue to run until a leak is discovered.

Cell	Patch	Solution	Comments
1A	Viton <sup>®</sup> B Foam + Viton <sup>®</sup> Sealant + RTV Sealant	12 hours NaOH >48 hours HNO <sub>3</sub>	No leaks. NaOH solution discolored slightly. Black flakes in bottom of beaker
1B	Viton <sup>®</sup> B Foam + Viton <sup>®</sup> Sealant + Liquid Metal Paste	12 hours NaOH >48 hours HNO <sub>3</sub>	No leaks. NaOH solution discolored slightly. Black flakes in bottom of beaker
2A	Viton <sup>®</sup> Sealant	12 hours NaOH >48 hours HNO <sub>3</sub>	No leaks. NaOH solution discolored.
2B	Viton <sup>®</sup> A Foam + Viton <sup>®</sup> Sealant	12 hours NaOH >48 hours HNO <sub>3</sub>	No leaks. NaOH solution discolored.
3A	Viton <sup>®</sup> B Foam + Viton <sup>®</sup> Sealant + RTV Sealant	12 hours NaOH	No leaks. NaOH solution discolored slightly. Black flakes in bottom after NaOH exposures, not seen during HNO <sub>3</sub> exposure. Temperature remained at 70°C.
		12 hours NaOH	
		14 hours HNO <sub>3</sub>	
		4 hours HNO <sub>3</sub>	
3B	Viton <sup>®</sup> B Foam + Viton <sup>®</sup> Sealant + Liquid Metal Paste	12 hours NaOH	No leaks. NaOH solution discolored slightly. Black flakes in bottom after NaOH exposures, not seen during HNO <sub>3</sub> exposure. Temperature remained at $70^{\circ}$ .
		12 hours NaOH	
		14 hours HNO <sub>3</sub>	
		4 hours HNO <sub>3</sub>	
4A	Viton <sup>®</sup> Sealant	12 hours NaOH	No leaks. NaOH solution discolored. Black flakes in bottom. Loss of adhesion of sealant on interior surface.
		12 hours NaOH	
		14 hours HNO <sub>3</sub>	
		4 hours HNO <sub>3</sub>	
4B	Viton <sup>®</sup> A Foam + Viton <sup>®</sup> Sealant	12 hours NaOH	No leaks. NaOH solution discolored. Black flakes in bottom. Loss of adhesion on interior surface.
		12 hours NaOH	
		14 hours HNO <sub>3</sub>	
		4 hours HNO <sub>3</sub>	

## **Table 5: Performance Testing Experimental Matrix**

## 5 RECOMMENDED REPAIR PATCH

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The recommendation is based upon the results of the exposure testing and continuing performance testing graded against the functional requirements. All of the measurements for the recommended application are approximate and are not subject to dimensional tolerances due to the nature of the materials being used. The recommendation for the stop-leak multi-layered approach is shown in Figure 7. The repair patch consists of a stainless steel backing plate with a  $\frac{1}{2}$ " Viton<sup>®</sup> B Foam, followed by Pelseal 2012 Viton<sup>®</sup> sealant, and a centered plug of RTV sealant. The foam

has 1/4" - 1/2" thick layer of Pelseal 2012 sealant troweled onto the foam surface. The material will be applied with a bevel, i.e. the material will be thicker towards the top since the sealant creeps over time when held in a vertical position. However, 4" of foam area is left uncovered along the perimeter of the foam surface. This allows for gauging of appropriate pressure application during remote delivery of the patch. As the sealant oozes out of the sides of the package, a decision will be made as to when enough pressure has been applied. A small 1" diameter x 4" thick layer of RTV silicone sealant in a ring configuration shall be applied on the surface of the Viton<sup>®</sup> sealant in the center of the package. This is meant for added resistance to the initial caustic flush during evaporator operation.

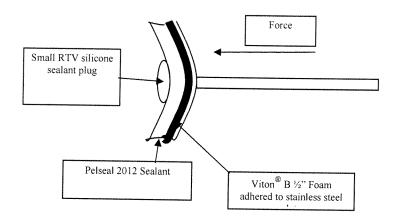


Figure 7: Recommended Repair Patch for 8.5E

### 6 CHALLENGES AND APPLICABILITY

The recommended repair patch method is based upon extensive short-term testing and conservative experimental evaluation in order to meet immediate facility needs. Several challenges are foreseen during the implementation and use of the repair patch during evaporator operation. A summary of the challenges and the ways in which they are addressed are shown in Table 6.

Challenges	Response
The time of effective leak seal cannot be estimated with the limited testing completed for this recommendation.	The lead surveillance testing will be continued until a leaksite is discovered. Post leakage analysis will be done on the lead surveillance test cans.
	The material can be reapplied with minor adaptations if failure is premature, but not necessarily early into evaporator restart.
The resistances to degradation when exposed to other organic materials known to be aggressive towards the candidate materials are unknown.	The limited surface area of exposure to the evaporator contents due to the small size of the penetration should improve the lifetime of the patch.
The head pressure the patch will be subject to has not been simulated, and consequent adhesion issues have not been addressed	Constant horizontal pressure applied by the delivery rig to the repair package should minimize the influence of the head pressure.
The surface of the tank was not perfectly prepared, e.g. sandblasting, as per vendor recommendations	Constant horizontal pressure applied by the delivery rig to the repair package assists with possibly poor

Table 6: Summary of Challenges with Repair Patch

Challenges	Response
	adhesion.
The corrosion mechanism for the leaksite is unknown, but has been speculated upon (in corrosion evaluation section)	Possible corrosion mechanisms discussed earlier suggest that the most aggressive conditions are in this section of the evaporator.

# 7 CONCLUSIONS AND RECOMMENDED PATH FORWARD

Additional investigations and evaluation of alternative sealing options are recommended for this application and others for continued and future operations. This may be of particular interest for applications in which external pressure cannot be applied or maintained. Materials capable of sealing against pressurized leaks, as well as being tolerant of less than ideal surface conditions may be required. Materials with shorter cure times, greater chemical and/or radiation resistance, or improved mechanical properties may also be needed.

### **REFERENCES**

<sup>1</sup> "Selection Criteria for Type 300 Series Stainless Steels," SW 301 M, Materials of Construction Standard Engineering Specification

<sup>2</sup> W. Hinz, E-Mail To: K. Subramanian, "221-F 8.5 Evaporator Leak - Visual Observation," August 13, 2003.

<sup>3</sup> From: W. P. Bebbington, To: J. W. Wade, "Effect of Nitric Acid Concentration on Evaporator Corrosion," SEP(N)-CAN-23-6, July 9, 1962.

<sup>4</sup> R. Eubanks, E-Mail To: K. Subramanian, "8.5E Low Activity Waste Evaporator Operating Conditions," August 7, 2003.

<sup>5</sup> Gray, J. "Tributyl Phosphate in Solids (U)," SRT-ATS-2003-00041, July 2003.

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