Contract No:

This document was prepared in conjunction with work accomplished under Contract No. DE-AC09-08SR22470 with the U.S. Department of Energy (DOE) Office of Environmental Management (EM).

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SDU6 Interior Liner Testing & Evaluation

T. E. Skidmore

September 2016

SRNL-STI-2016-00568, Revision 0

SRNL.DOE.GOV

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Printed in the United States of America

Prepared for
U.S. Department of Energy

Keywords: Saltstone, polymer, liner

Retention: *Permanent*

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ACKNOWLEDGEMENTS

The author acknowledges the assistance of several individuals for this task. Liner sample immersion was performed by SREL personnel (J. C. Seaman, J. P. Cochran and E. R. Dorward) with oversight and assistance from Steven Simner (SRR/Closure & Disposal Assessment). Project/design input by Sergio Mazul, Noel Chapman, J. P. Thompson and Thomas Brooks is gratefully acknowledged, as well as input from liner manufacturer representatives. Tensile and lap-shear testing was performed by Jim Wilderman (SRNL/Material Science & Technology), with samples cutting performed by the SRNL/723-A Machine Shop. Sample photography was performed by Robbie Garritano and Lin Thacker (SRNL/Material Science & Technology).

EXECUTIVE SUMMARY

Two liner materials (Marseal® M-3500 and REMA Chemoline® 4CN) proposed for use as a liner inside the Saltstone Disposal Unit 6 (SDU6) were subjected to specific ASTM tests (tensile and lap-shear) after immersion in 50% and 100% simulant solutions for 1000 hours at the Savannah River Ecology Laboratory. Both liner materials exhibited good resistance to the simulant chemistry, at least based on the tests performed and the test duration/conditions imposed. In lap-shear tests, both materials failed in the base material rather than peeling apart, confirming good adhesion. The REMA 4CN bromobutyl elastomer showed superior bonding characteristics and absence of warping or delamination at the conditions tested. The Marseal M-3500 material (PVC/EVA blend with polyester reinforcement) exhibited deformation and debonding in some locations. The cause of the deformation and delamination observed in the Marseal M-3500 material is not fully known, but possibly attributed to thermomechanical stress at immersion temperatures, and the thermoplastic nature of the material. The immersion temperature (68 °C) is slightly greater than the maximum use temperature limit quoted for the Marseal M-3500 liner (65 °C), though the basis for the service limit is unknown. The testing performed was limited in scope and only for these two liner materials. These tests were primarily performed to screen for severe incompatibility or short-term degradation in Saltstone bleedwater simulants at bounding solution temperatures. Additional testing is recommended to assess long-term performance and the overall service life of the liner.

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LIST OF ABBREVIATIONS AND ACRONYMS

ASTM	American Society for Testing of Materials
CRC	Chemical Resistant Coating
CSPE	Chlorosulfonated polyethylene
CSSX	Caustic Side Solvent Extraction
DOE	Department of Energy
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetry
ECTFE	Ethylene-chlorotrifluoroethylene
EIA	Ethylene interpolymer alloy
EPA	Environmental Protection Agency
EPDM	Ethylene-propylene diene monomer
EPR	Ethylene-propylene copolymer (rubber)
ESCR	Environmental stress-cracking resistance
ETFE	Ethylene-tetrafluoroethylene
EVA	Ethylene-vinyl acetate
FEP	Fluorinated ethylene-propylene
FML	Flexible membrane liner
FTIR	Fourier Transform Infra-Red
G Value	Molecules per 100 eV
Gray	International Unit of ionizing radiation absorbed, 1 Gy = 100 rad
GRI	Geosynthetic Research Institute
HDPE	High density polyethylene
ICRI	International Concrete Repair Institute
IIR	isobutylene isoprene rubber
KEE	Ketone ethylene ester
LDPE	Low density polyethylene
LLDPE	Linear-low-density polyethylene
LTLS	Leak-tight liner system
NBR	Acrylonitrile-butadiene rubber (nitrile)
PCTFE	polychlorotrifluoroethylene
PFA	perfluoroalkoxy
pPVC	Plasticized polyvinyl chloride
PTFE	polytetrafluoroethylene
PVC	Polyvinyl chloride
PVDF	Polyvinylidene fluoride
Rad	Unit of ionizing radiation energy absorbed (1 rad = 100 erg/g)
SBR	Styrene-butadiene rubber
SDU	Saltstone Disposal Unit
SREL	Savannah River Ecology Laboratory
SRNL	Savannah River National Laboratory
SRR	Savannah River Remediation
SRS	Savannah River Site
SWPF	Salt Waste Processing Facility
T_g	Glass transition temperature
T_m	Melting temperature
TBP	Tri-butyl phosphate
TTS	Time-Temperature Superposition

1.0 BACKGROUND & INTRODUCTION

The Saltstone Disposal Unit 6 (SDU6) is a circular concrete tank approximately 375 feet in diameter by 43 feet high, contains 208 roof support columns/footings, with a total capacity of 32 million gallons. The column footings are 5 foot square by 18 inches high. The columns are 2 feet in diameter. After accounting for cold cap requirements, the SDU6 will be capable of dispositioning approximately 30 million gallons of low-level contaminated grout [1]. The SDU6 design (Figure 1) is based on the Syracuse, NY Westcott Reservoir design [2]. In comparison, the current active SDUs (SDU-3 and SDU-5) consist of two cells nominally 150 feet diameter by 22 feet high.

SDU6 was initially required to meet ACI 350.1 requirements for water tightness, but without coatings or linings per TR&C documents [1, 3, 4]. The ACI 350.1 acceptance criterion consists of Part 1 (qualitative) and Part 2 (quantitative) tests. Part 1 criterion is the exterior surfaces shall not have moisture that can be picked up on a dry hand. Part 1 allows for wet areas on top of the wall footing but does not allow for observation of flowing water. Part 2 is a measureable loss criterion, which, if a particular criterion is specified, is defined as the drop in the water surface not exceeding 1/8 inch in 72 hours, measured within 1/16” and compensated for temperature and evaporation. Unless otherwise specified, coatings shall not be applied until after the hydrostatic tightness testing has been completed. Liners that are mechanically locked to the surface during the placement of the concrete shall be installed before the hydrostatic tightness testing [4].

Per project specifications, the water tightness acceptance criteria was A) no measureable loss (ACI 350.1) and B) no observation of traces of dye, either as observation of dye color by the naked eye or evidence of dye fluorescence not documented in the baseline, at any location exterior to the tank, up to the wetted level of the tank [1, 3].

SDU6 hydrostatic testing required approximately one month to fill (41 feet), completing on November 3, 2015. After waiting the required time period, the SDU-6 structure passed the no measureable loss testing and the walls exhibited no signs of dye or dampness, but the floor slab failed the dye tracer test and ACI 350.1 Part 1 as flowing water was observed at several locations (i.e. primarily between the floor slab and upper mudmat, and between the upper mudmat and external HDPE liner).

Draining the SDU6 began on November 11, 2015. Upon draining of the tank, cracks were observed on the interior tank floor (Figure 2). Subsequent investigation was performed by the U.S. Army Engineer Research and Development Center, Geotechnical and Structures Laboratory (ERDC-GSL) [5]. This evaluation, involving core sampling of the floor in five locations and petrographic analysis, revealed that several cracks extend completely through the floor slab, providing a pathway for water (and future Saltstone bleedwater) to reach the polyethylene barrier and work its way outward to the vault perimeter. The crack footage was estimated at ~10,000 linear feet. Similar cracking was also observed on the roof slab. A Nonconformance Report (2016-NCR-15-DZC-0005) was issued documenting that the floor slab cracks combined with the failure to pass leak tightness test yield SDU 6 indeterminate in meeting structural, leak tightness and Performance Assessment (PA)/SA (Special Analysis) requirements.

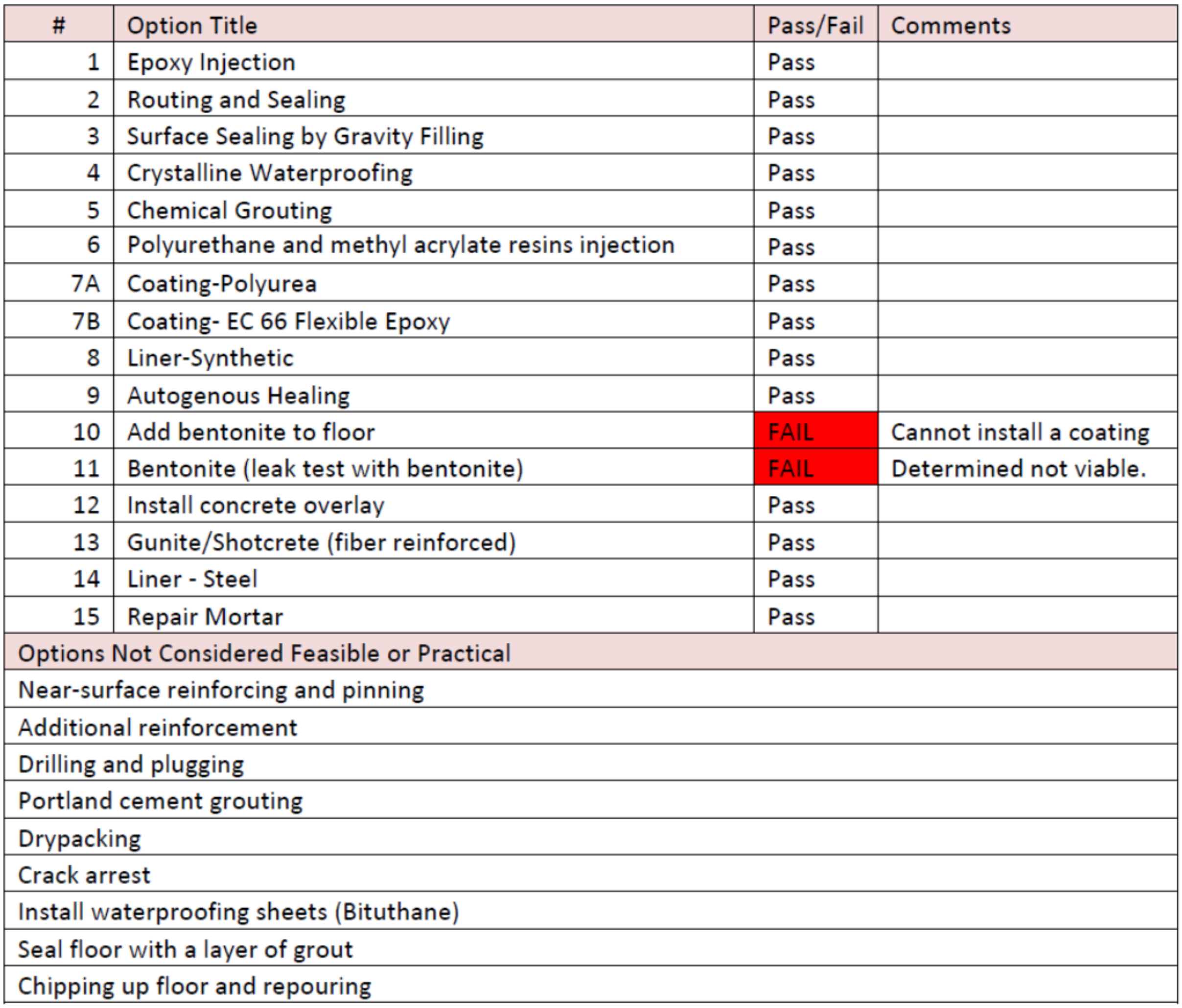
In March 2016, a SEE (Systems Engineering Evaluation) was performed to evaluate options for returning the SDU6 vault to compliance with meeting project leaktight requirements [6]. The SEE team consisted of personnel from several different organizations and disciplines, including DOE, CH2M, SRR/Project Management, Engineering, Design Services, SRNL and Construction. A separate external SME review was also conducted with concrete and coatings/linings experts from the U.S. Army ERDC-GSL and Bechtel.

The SEE team considered several options for bringing the SDU6 into leaktight compliance. The options considered are listed in Table 1 [6]. A Chemical Resistant Coating (CRC) system (EC-66 flexible epoxy, Versiflex/Blome International) was already specified for the SDU6 interior to protect the concrete from chemical degradation (principally sulfate attack), but the SDU6 was initially required to meet leaktightness without coatings or linings. The EC-66 coating system has been used in previous SDUs, either as a standalone coating or as a flexible finish coat over a TL-45 mat-reinforced epoxy novolac lining system.

Figure 1. Overview of current SDUs (vaults) in SRS Z-Area [7]

Figure 2. Interior view of SDU6 floor cracking and initial crack sealing attempts [8]

Table 1. Options Considered for Achieving SDU6 Leak-Tightness Requirements [6]



#	Option Title	Pass/Fail	Comments
1	Epoxy Injection	Pass	
2	Routing and Sealing	Pass	
3	Surface Sealing by Gravity Filling	Pass	
4	Crystalline Waterproofing	Pass	
5	Chemical Grouting	Pass	
6	Polyurethane and methyl acrylate resins injection	Pass	
7A	Coating-Polyurea	Pass	
7B	Coating- EC 66 Flexible Epoxy	Pass	
8	Liner-Synthetic	Pass	
9	Autogenous Healing	Pass	
10	Add bentonite to floor	🟥 FAIL	Cannot install a coating
11	Bentonite (leak test with bentonite)	🟥 FAIL	Determined not viable.
12	Install concrete overlay	Pass	
13	Gunite/Shotcrete (fiber reinforced)	Pass	
14	Liner - Steel	Pass	
15	Repair Mortar	Pass	
Options Not Considered Feasible or Practical			
Near-surface reinforcing and pinning			
Additional reinforcement			
Drilling and plugging			
Portland cement grouting			
Drypacking			
Crack arrest			
Install waterproofing sheets (Bituthane)			
Seal floor with a layer of grout			
Chipping up floor and repouring			

Based on consideration of all requirements and project criteria, including installed cost, time/schedule impact, installation feasibility, likelihood of success (i.e. meeting leaktight requirements), service life (6 year active operations, 25 year service life) and other factors, the SEE team recommended two primary options, namely: 1) epoxy injection/crack repair and 2) installation of synthetic liner [6]. Some options (e.g. steel lining) were expected to be highly successful, but were more difficult to install or would significantly impact project cost/schedule. Other options were less expensive or easier to deploy but were judged less likely to be successful.

Of the two primary options, epoxy injection methods were considered challenging based on previous SDU6 project experience during initial crack evaluation, the crack linear footage, repair verification difficulty and feedback from concrete SMEs and concrete crack repair vendors. Therefore, the SEE team recommended installation of a synthetic liner as the preferred option. External SME review (P.R. Nau/Bechtel) similarly concluded that installation of a bonded sheet lining was likely the most viable option within reasonable cost/time constraints, though no specific products were recommended [8].

A synthetic liner was preferred over the use of a liquid-applied coating system (such as the specified EC-66 flexible epoxy or other coatings) due to the variables involved. A particular concern was possible movement of existing cracks or opening of new cracks during SDU6 operations, causing coating failure. Although elastomeric coatings can tolerate some crack movement, the crack-bridging capability of the proposed coatings is not well-established. In addition, the specified coating was principally intended to protect the concrete from chemical attack, not to ensure leaktightness. The coating vendors could not provide assurance that the coating systems alone would provide leaktightness for the duration required (active operations: 6 years, service life: 25 years).

An option considered was the incorporation of a fabric reinforcement layer within either the EC-66 flexible epoxy or an alternative coating (such as Envirolastic® AR425 polyurea, Sherwin-Williams) to improve crack-bridging capability and structural integrity of the coating(s) on the tank floor. The polyurea coating had previously been proposed as an alternative to the flexible epoxy EC-66 coating for concrete protection. The use of fabric or geotextile-reinforced polyurea coatings is becoming a more common approach for primary and secondary containment applications such as wastewater structure linings and geomembrane applications [9, 10].

Though fabric-reinforced coatings may be sufficient to maintain leaktightness of the SDU6 with existing cracks, particularly without additional crack movement, the SEE team and external SMEs considered that installation of a synthetic liner was preferred and is less reliant on the substrate condition. Additional consideration of fabric or geotextile-reinforced coatings may be warranted for future SDUs, particularly if floor cracking can be mitigated.

Several generic liner materials were considered for the SDU6 application. A detailed review is beyond the scope of this report but generic types are briefly discussed. These are also briefly discussed in Reference 47. Such materials are often described as flexible membrane liners (FMLs), particularly for landfill liners and geomembranes. Polymers used for pond liners, geomembranes, tank linings and roofing membranes generally fall into the following types, with specific properties, strengths and limitations [11-13]. Liquid-applied coatings used for tank lining purposes are not discussed here.

Thermoplastics or modified thermoplastics

- high-density polyethylene (HDPE)
- linear-low density polyethylene (LLDPE)
- polypropylene (PP)
- polyvinyl chloride, typically plasticized (pPVC)
- pPVC/EVA (ethylene vinyl acetate) blends or EVA/KEE, EIA (ethylene interpolymer alloy)
- TPO (thermoplastic polyolefin) or TPE (thermoplastic elastomer)
- fluoropolymers such as Teflon (PTFE, FEP, PFA), PVDF, ETFE, ECTFE and PCTFE

Thermosetting elastomers

- butyl (and halogenated varieties, bromobutyl and chlorobutyl)
- ethylene-propylene diene monomer (EPDM)
- natural rubber (isoprene)
- chlorosulfonated polyethylene (CSPE or CSM, Hypalon® or similar)
- polychloroprene (neoprene)
- fluoroelastomer (Viton® or similar)
- nitrile rubber (NBR, acrylonitrile-butadiene rubber)
- SBR (styrene-butadiene rubber)

Of the thermoplastic or modified thermoplastic liners, HDPE (high-density polyethylene) is the most widely used and most well-studied, being commonly used in landfill or hazardous waste liner and geomembrane applications. HDPE has been extensively studied for such applications by the Geosynthetic Research Institute (GRI) and other agencies and is known to provide many years of service, HDPE has also been specified for SDU exterior liner applications as a hydraulic barrier and to protect concrete from groundwater degradation, with long-term performance reviewed [14, 15].

HDPE has been extensively studied for many applications, though it has been less studied in high pH solutions at elevated temperature. This is primarily because most geomembrane applications do not involve aggressive chemicals or elevated temperatures. Some studies have indicated possible sensitivity to environmental stress-cracking (ESC) in high pH solutions at elevated temperature, though others have shown pH independence [16, 17].

The ESCR (environmental stress-cracking resistance) of HDPE is a primary concern in geomembrane/liner applications. The chemistry of the Saltstone bleedwater is not particularly aggressive to many polymeric materials, but the combination of high pH, radiation, chemistry, elevated temperature and stress is not trivial. In some cases, geomembranes and liners based on LLDPE (linear-low density polyethylene) or TPO (thermoplastic polyolefin) provide improved flexibility and performance over that of HDPE, particularly with regard to ESCR [11-13].

Fluoropolymers such as Teflon (PTFE, FEP, PFA), PVDF, ETFE, ECTFE and PCTFE offer superior chemical resistance but are much more expensive and not practical for large-scale tank linings. As with HDPE, these polymers are not particularly flexible and require specialized bonding equipment. The radiation resistance of some fluorinated polymers (particularly PTFE) is also known to be relatively low. Other types, such as PVDF, can be sensitive to high pH solutions, particularly at elevated temperatures.

Polypropylene (PP) offers excellent chemical resistance and is less prone to environmental stress-cracking than HDPE but is less resistant to ionizing radiation than most other thermoplastic types. Many geomembrane and roofing applications now use ethylene blends or copolymers known collectively as ethylene interpolymer alloy (EIA), some of which are blended with Ketone Ethylene Ester (KEE). Examples of these products are the XR series of geomembranes from Seaman Corporation [18].

Plasticized polyvinyl chloride (pPVC) and PVC/EVA (ethylene-vinyl acetate) polymers are resistant to many chemicals, including high pH solutions. Being principally amorphous (non-crystalline), PVC-based materials are sensitive to certain organics and solvents. The concentrations of such species in the Saltstone bleedwater are likely very low, but could potentially affect PVC-based liners over long service periods. If organic concentration increases in future SDU feed streams (such as from the SWPF), additional evaluation may be needed. A major advantage of the EIA and PVC/EVA polymers compared to traditional plasticized PVC (pPVC) is the lack of low-molecular weight plasticizers that can migrate over time, resulting in polymer embrittlement.

Of the thermosetting elastomer options, each type poses unique advantages and potential disadvantages, depending on actual conditions, property retention needed and other requirements. Several elastomeric liners would likely work in the SDU6 application. The generic advantage of elastomeric liners is their superior resiliency and flexibility, allowing for easier installation on many substrates. Elastomers also do not suffer from environmental stress-cracking (ESC) as does HDPE. Stiffer polymers may be easier to install around sharper edges or interfaces, particularly if the materials can be thermally pre-formed to the necessary shape (such as column footings).

Butyl rubber (and halogenated varieties, bromobutyl and chlorobutyl) provides broad chemical resistance and excellent (low) gas permeability, with good resistance to aging, ozone and thermo-oxidation. A limitation of butyl rubber (isobutylene-isoprene) is lower resistance to ionizing radiation, as compared to most other elastomers [19-21]. Halogenated versions (chlorobutyl, bromobutyl) tend to improve the cure rate, reversion resistance and co-vulcanisation with other diene rubbers, as well as improving chemical resistance and fire retardancy. Some references also suggest that the radiation resistance of halobutyl is superior to that of non-halogenated butyl rubber [22-23].

EPDM (ethylene-propylene diene monomer) is widely used in roofing membrane and pond liner/landfill membrane applications, with excellent resistance to aging/ozone, thermo-oxidation and a broad range of chemicals, including high pH solutions. Like butyl rubber, EPDM is resistant to many chemicals, but it has superior resistance to ionizing radiation compared to most elastomers, being used for nuclear cable insulations (as well as EPR, ethylene-propylene copolymer). EPDM would likely be a viable liner candidate for the SDU6 application, though testing would be needed to confirm this assumption.

Natural rubber (polyisoprene) has excellent resistance to ionizing radiation and abrasion, but is less resistant to certain chemicals and has limited resistance to aging, ozone and thermo-oxidation without special additives. Similarly, nitrile rubber (NBR, acrylonitrile-butadiene rubber) has excellent resistance to many hydrocarbon compounds (fuels, oils) and to some aqueous solutions, but it also has limited resistance to aging, ozone and thermo-oxidation.

Chlorosulfonated polyethylene (CSPE or CSM, Hypalon® or similar) is a chemically-modified thermosetting polyethylene that provides excellent resistance to a broad range of chemicals, ionizing radiation and thermo-oxidation. CSPE/CSM has been widely used in nuclear power cable jackets, chemical transfer hose, roofing and pond liner/landfill membranes and geosynthetics. DuPont, which invented and produced Hypalon® CSPE since the 1950s, discontinued U.S. production in 2010 due to competition with other materials as well as regulatory issues related to formulation and combustion gas toxicity [24]. CSPE is still available from Japan and other sources. CSPE would likely be a viable candidate for the SDU6 liner due to combined properties, though availability and formulation aspects would have to be evaluated in more detail.

Polychloroprene (neoprene) is reasonably resistant to aging/thermo-oxidation and a broad range of chemicals (limited in acids), as well has having moderate resistance to ionizing radiation. A limitation of neoprene, similar to PVC, is that it has a very high chloride concentration, which can contribute to corrosion and possible HCl evolution if the polymer degrades. Fluoroelastomers such as Viton® offer superior resistance to many chemicals, including many organics and acids, but are much more expensive than other elastomers and most formulations have sensitivity to high pH solutions, particularly at elevated temperature. Special grades are available for high pH solutions.

The specific radiation resistance and off-gas behavior of coatings and lining materials in the SDU6 application is beyond the scope of this report. All polymers will generate gas as a result of thermal or radiolytic degradation. The amount and type of gas generated depends on several factors such as polymer type, specific formulation, oxygen, temperature, radiation dose and dose rate. Certain polymers (HDPE) primarily generate flammable gases such as hydrogen, which is a safety concern. Other polymers, particularly PVC, primarily generate HCl gas, which is not flammable but is corrosive in the presence of moisture [25-26]. Dehydrochlorination of PVC does not significantly occur <100 °C, though some references suggest that it can occur as low as 80 °C [27, 28]. This is still above the 68 °C immersion temperature used for testing liner materials. The threshold radiation dose for HCl gas generation from PVC is not well-defined, but has been observed at doses of 1-3 Mrad [25]. Plasticizers tend to reduce HCl evolution from PVC, but if plasticizer migration occurs, HCl evolution may increase. Halobutyl rubbers may generate HCl or HBr gases, though the halogen content is far less than that of PVC.

The impact of gas generation from the liner during the SDU service life is not likely significant but may require separate evaluation. Flammable gas monitoring from the waste form is already part of the SDU design. Halogenated, acidic gases (HCl, HBr) would likely be neutralized in a high pH aqueous environment, so this is also not likely a significant concern.

SRR performed a review of readily-available liner alternatives and identified several for consideration [47]. Many of these were eliminated for various reasons, such as principal use in secondary (not primary) containment, inadequate or unknown chemical resistance, temperature limitations or general absence of data. Some products, while possibly viable, were principally used for roofing or other non-immersion applications.

The options reviewed were narrowed down to the two following materials: 1) REMA Chemoline® 4CN (bromobutyl rubber, REMA Tip Top North America, Inc., Madison GA) and 2) Marseal® M-3500 (EVA/PVC blend, Blair Rubber Co., Seville, OH). Marseal M-3500 was initially discussed during the SEE evaluation [6]. Both liners were considered to have reasonable resistance to the Saltstone bleedwater chemistry and service temperatures involved. The Chemoline® 4CN material is used as primary containment in many corrosive environments [29]. The Marseal product line (formerly Martin Rubber Company) is more commonly used for secondary containment applications, though it has been used for primary containment [30]. Blair Rubber also offers primary containment liners based on halobutyl and other elastomers, similar to the Chemoline 4CN.

Marseal® M-7000 and M-8000 liners (EPDM-based) were also considered possible candidates based on the known resistance of EPDM to high pH solutions, thermo-oxidation and ionizing radiation. However, the manufacturer (Blair Rubber) could not recommend these products due to concern over certain species in the Saltstone bleedwater chemistry. The basis for this concern is unclear. It may be that some species, if present in high concentrations or saturated solutions, could indeed degrade EPDM elastomer. But the concentrations within the bleedwater are much lower and likely much less aggressive. However, due to vendor concerns, the Marseal® 7000/8000 materials were not considered further. If additional SDU liner tests are planned, inclusion of an EPDM liner such as Blair Rubber's Marseal®7000/8000 or Enduraflex™ EPDM or similar products is suggested.

2.0 LINING TESTS AND METHODOLOGY

SRR previously contracted with CUA (Catholic University of America) and the Savannah River Ecology Laboratory (SREL) to perform immersion exposure tests on two coatings used in previous SDU designs, one being a mat-reinforced epoxy novolac (TL45S, Blome International) and one being the EC-66 flexible epoxy [31]. Another coating (Envirolastic® AR425 polyurea, Sherwin-Williams) was later included in SREL immersion tests, as this coating was discussed as a possible alternative lining during SDU6 specification development.

These tests essentially involved ASTM D6943-type exposures to Saltstone bleedwater simulants at a bounding temperature of 68°C. This temperature was previously calculated as a bounding grout/bleedwater temperature in the SDU2 design [32-34]. For the SDU6 design, variation in grout fill volume, pouring rates and other operational aspects may reduce this temperature, but such values have not been confirmed as of the writing of this document. Lower temperatures, if present, would be less challenging to coatings and linings.

Liner samples of the REMA Chemoline 4CN and Marseal 3500 liner materials were submitted by SRR to the SREL for 1000-hour immersion exposures at 68 °C in two different versions of an S1 simulant (designated as 50% and 100% solutions), Table 1. The chemicals used for the simulant make-up include: NaOH, KOH, $Al(NO_3)_3$, $NaNO_2$, Na_2SO_4, NaCl, $NaCO_3$, Na_3PO_4 and ammonium oxalate ($(NH_4)_2C_2O_4$). The simulant solution density (100%) is approximately 1.3 g/cc [35].

Description of the experimental set-up and sample observations is documented in Reference 35. The 1000 hour exposure time is an optional test period in ASTM D6943-15, Standard Practice for Immersion Testing of Industrial Protective Coatings and Linings, and is consistent with previous estimates of time at maximum grout/bleedwater temperature in SDU2 [32-34]. It is emphasized that there is nothing magical about a particular immersion period. Shorter exposures (such as 72 hours) might only be needed for secondary containment purposes and are likely sufficient to identify significant short-term incompatibility. However, longer exposures are likely needed for service life evaluation.

It is noted that the simulant solutions did not include any organics. Organics are known to significantly degrade polymeric materials. Significant organics are not expected in the feed streams to SDU6. However, if organic concentrations in Saltstone bleedwater increases due to carryover from future SWPF waste transfers or other sources, organic content may be of more concern. Trace amounts of antifoaming agents in the Saltstone process (now silicone fluid, previously tributyl phosphate, TBP) may also exist. Silicone-based fluids are benign to most polymeric materials (with exception of silicone elastomers), whereas TBP is a known wetting agent and surfactant/stress-cracking agent for many polymers, particularly amorphous polymers such as polyvinyl chloride (PVC). The effect of low concentration of such species on the liner is likely minimal, though cumulative effects are possible. Future tests, if planned, should include organic species anticipated in the process.

Following 1000-hour simulant exposures at SREL, baseline (non-immersed) and immersed samples were submitted to SRNL/Materials Science & Technology (MS&T) for additional testing and evaluation. A list of sample designations and descriptions is provided in Table 2 [35].

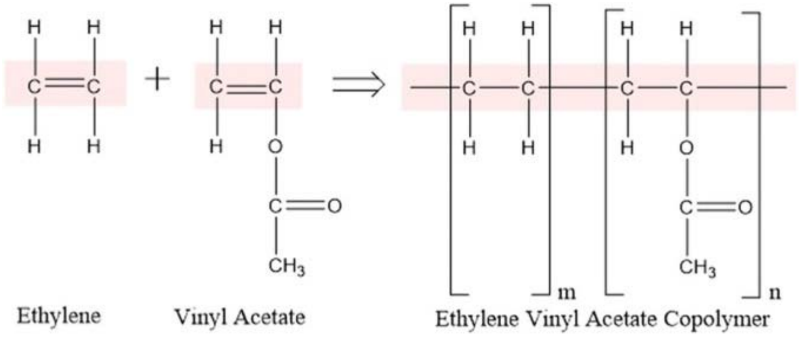
Table 1: Saltstone Bleedwater Simulant Salt Solution (S1) and Rationale [35]

Species	Conc. (M)	Rationale
Na^+	6.73	Maximum [Na^+] per SWPF WAC is 7.0 M; however, total cation/anion inventory had to be reduced to avoid precipitates
Al^{3+}	0.22	Approximates historically measured [Al^{3+}] in Tank 50
K^+	0.06	Maximum [K^+] per SWPF WAC
OH^-	2.30	Maximum [OH^-] per SWPF WAC
NO_3^-	2.35	Approximates historically measured [NO^{3-}] (average) in Tank 50; meets corrosion inhibitor requirements
NO_2^-	0.90	Nitrite is added at approximately double the upper concentration recorded in Tank 50 since Ref. 41 indicates higher nitrite concentrations in future salt batches from the Tank Farm
CO_3^{2-}	0.20	Approximates historically measured [CO_3^{2-}] in Tank 50
SO_4^{2-}	0.18	Maximum [SO_4^{2-}] per TF WAC
Cl^-	0.11	Maximum [Cl^-] per TF WAC
PO_4^{3-}	0.05	[PO_4^{2-}] is limited by solubility (refer to latter text on preparing simulants); concentration higher than maximum measured in Tank 50
$C_2O_4^{2-}$	0.01	Approximates historically measured [$C_2O_4^{2-}$] in Tank 50

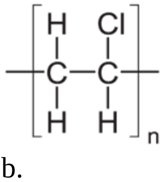
Table 2. List of SREL Liner Sample Designations and Description [35]

Sample Code	Sample Description
1SREL-LS-MAR-100-052416	MARSEAL-3500, with seam, 100% S1 solution
2SREL-LS-MAR-050-052416	MARSEAL-3500, with seam, 50% S1 solution
3SREL-LI-MAR-100-052416	MARSEAL-3500, intact (baseline), 100% S1 solution
4SREL-LI-MAR-050-052416	MARSEAL-3500, intact (baseline), 50% S1 solution
5SREL-LS-REM-100-052416	Chemoline 4CN, with seam, 100% S1 solution
6SREL-LS-REM-050-052416	Chemoline 4CN, with seam, 50% S1 solution
7SREL-LI-REM-100-052416	Chemoline 4CN, intact (baseline), 100% S1 solution
8SREL-LI-REM-050-052416	Chemoline 4CN, intact (baseline), 50% S1 solution
9SREL-CI-MRU-050-052416	MARSEAL-2000, corner intact, 50% S1 solution
10SREL-CI-MRU-100-052416	MARSEAL-2000, corner intact, 100% S1 solution
11SREL-CS-MRU-050-052416	MARSEAL-2000, corner with seam, 50% S1 solution
12SREL-CS-MRU-100-052416	MARSEAL-2000, corner with seam, 100% S1 solution
13SREL-LB-MAR-100-053116	MARSEAL-3500, bonded to coated concrete block, 100% S1 solution
14SREL-LB-MAR-050-053116	MARSEAL-3500, bonded to coated concrete block, 50% S1 solution
15SREL-LB-REM-100-053116	Chemoline 4CN, bonded to coated concrete block, 100% S1 solution
16SREL-LB-REM-050-053116	Chemoline 4CN, bonded to coated concrete block, 50% S1 solution

The Marseal® M-3500 liner material is a 75-mil (1.91 mm) PVC/EVA (Elvaloy®, DuPont) polymer blend, with polyester reinforcement. The general polymer structure of PVC and EVA polymers is shown in Figure 3a and 3b. The Marseal® 2000 corner material is a 60-mil (1.524 mm) PVC / EVA polymer blend. The Marseal 2000 material was proposed by the vendor (Blair Rubber) for column footings due to squared edges and preformability. The increased stiffness of the Marseal 2000 samples suggests a different PVC to EVA ratio, with decreased flexibility attributed to lower amounts of EVA. EVA copolymers can significantly vary in the ethylene to vinyl acetate (VA) ratio with most products containing 10-40%, though lower and higher VA contents are possible [36]. The final PVC/EVA blend can also vary in PVC to EVA ratio.



3a.

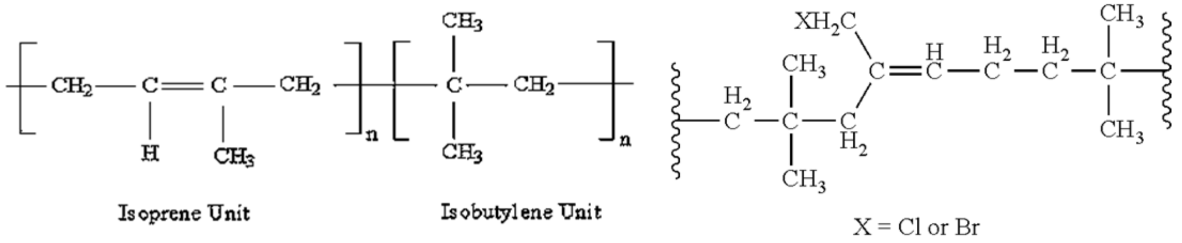


3b.

Figures 3a (ethylene-vinyl acetate copolymer) and 3b (polyvinyl chloride)

The REMA Chemoline® 4CN material is a 3 mm (118 mil) thick pre-vulcanized bromobutyl elastomeric liner. Butyl rubber (IIR) is a copolymer of isobutylene (98-99%) and isoprene (1-2%), commercialized in the early 1940s as an urgent synthetic replacement for natural rubber. To improve several properties such as the cure rate (increase), reversion resistance, co-vulcanization with other diene rubbers, chemical resistance and fire retardancy, butyl rubber is halogenated with chlorine or bromine to produce chlorobutyl and bromobutyl derivatives [37].

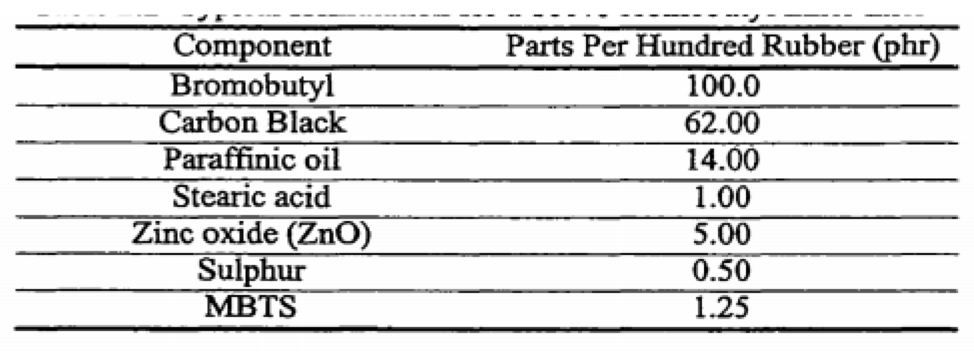
In chlorobutyl, the chlorine% is ~ 1.25% whereas bromobutyl might contain up to 2% bromine. The chemical formulas for butyl and halobutyl rubber are shown in Figures 4a and 4b. A typical bromobutyl rubber formulation is shown in Table 3 [37]. Typical properties of the liners are given in Table 4. These are not all-inclusive and direct comparison is difficult due to variation in test standards and available data. The specific formulations of the Marseal M-3500 and REMA 4CN liners are proprietary.



4a. 4b.

Figures 4a (butyl rubber structure) and 4b (halobutyl structure)

Table 3. Typical Bromobutyl Rubber formulation [37]



Component	Parts Per Hundred Rubber (phr)
Bromobutyl	100.0
Carbon Black	62.00
Paraffinic oil	14.00
Stearic acid	1.00
Zinc oxide (ZnO)	5.00
Sulphur	0.50
MBTS	1.25

Table 4. Liner Typical Property Comparison [29, 30]

Property	Method	Unit	Marseal M-3500	Chemoline 4CN
Durometer hardness	ASTM D2240 (ISO 868-2003)	A scale	85+/-5	50+/-5
Elongation at break	ASTM D412 (ISO 37-2005)	%	170	≥370
Tensile Strength	ASTM D412 (ISO 37-2005)	psi (MPa)	1500 (10.3)	725.2 (≥ 5)
Tear Strength	ASTM D624	lb/in	330	Not specified
Max service temperature	Not specified	°F/°C	150/65	185/85
Water Vapor Transmission/Permeability	ASTM E96 (DIN 53122)	Grains/h-ft2 (g/m2/day)	0.065	(0.04)

Note: The REMA 4CN material is typically tested to ISO and DIN standards, with the Marseal M-3500 product being tested to ASTM standards. Variation in test standards may result in different values/units.

Photographs of the liner samples received from SREL are given in Figures 1-35. Baseline (non-immersed) samples are shown in Figures 1-10. SRNL/MS&T witnessed removal of the samples from the SREL immersion baths, with crystalline precipitates noted to be present on all samples to some extent, but to a greater extent on the gray side of the REMA bromobutyl liner. This side has a visible texture which is presumably intended to aid in adhesion to the substrate. Removal of precipitates under running water was more difficult for these surfaces. The precipitates were not analyzed.

Upon initial examination, the liner materials were found significantly different in terms of flexibility/stiffness and bonding configuration. The Marseal M-3500 material was bonded using a simple lap joint, with approximately 2.75" overlap. The REMA 4CN material was bonded using a 45° splice joint, with approximately ¾" bond length. The bonded materials are presumed to have been bonded as per normal installation instructions provided by the manufacturer. SRNL/MS&T did not verify the bond configuration with the vendor. Specific bonding parameters (temperature, pressure, cement type) were not disclosed.

Several samples of the self-bonded Marseal M-3500 material exhibited regions of delamination and "puckering" to varying degrees on the as-received samples (Figures 12-13, 18-22). This was noted in the SREL test report to occur on day 35 of the immersion period [35]. It is unknown if this behavior occurred sooner, but the samples were inspected on a daily basis, including verification of sample chamber temperature (68 +/-2 °C). Similar behavior was also observed on the samples of Marseal M-3500 bonded to the Marseal® 2000 material (MRU samples), noted to occur on day 23 of the immersion period [35]. The delamination was observed to be slightly more severe for the 100% S1 solution samples than for the 50% S1 solution, with cause unknown. The Marseal M-3500 sample solutions were also noted to change color initially to a dark yellow and become a dark reddish brown over the course of testing, particularly in the 100% S1 solution. The cause of the discoloration was not investigated.

The Marseal 2000 (non-bonded) samples remained stiff and coiled when removed from their immersion containers (Figures 16-17). This is presumably due to the thermoplastic nature of the polymer blend, with limited flexibility and lower EVA content. Reheating of the material and flattening may restore the material to the original shape, but this was not attempted. Per SRR input, these materials were not tested for baseline or post-immersion properties.

Samples of the intact or non-bonded liner materials were tested per ASTM D412-15, Standard Test Methods for Vulcanized Rubber and Thermoplastic Elastomers [38]. An example of tensile samples die-stamped from the REMA 4CN material are shown in Figure 10. Similar samples were stamped from the Marseal M-3500 material. Bonded samples were tested per ASTM D6392-12, Standard Test Method for Determining the Integrity of Non-reinforced Geomembrane Seams Produced using Thermo-Fusion Methods [39]. Only the lap-shear test configuration was tested due to inability to perform T-peel tests. Lap-shear/bond joint test samples for Marseal M-3500 and REMA 4 CN materials are shown in Figures 8-9 (baseline) and 36-44 (immersed, pre-test).

ASTM D4437, Standard Practice for Non-destructive Testing (NDT) for Determining the Integrity of Seams used in Joining Flexible Polymeric Sheet Geomembranes, was used to evaluate the integrity of the liners bonded to a concrete block previously coated with EC-66 flexible epoxy [40]. This configuration (liner bonded to EC-66 epoxy) is proposed in the SDU6 design at the base of support columns and possibly other locations to provide additional sealing of the concrete surface around the base of the support columns. These samples are shown in Figures 23-35.

The ASTM D6392 and D4437 standards are explicitly written for geomembrane materials, not specifically for tank linings, but the principal methods are deemed sufficient for liner material comparison purposes. Only the point contact method was used per ASTM D4437, with a blunt screwdriver used around the edges of the bonded sheets to check for bond integrity.

Samples of the baseline and immersed liner sections (both intact and bonded) were prepared for testing. A minimum of 5 tensile samples were created for each liner section using a die punch meeting ASTM D412, Die D dimensions. Lap-shear strip samples were cut to a 1” width using a sharp cutting knife and a metal straight edge. The bromobutyl rubber material was slightly more difficult to cut than the Marseal M-3500 material. This may be attributed to variation in material properties and/or thickness (1.91 mm for Marseal M-3500 vs. 3 mm for the Chemoline 4CN material).

Photographs of the bonded lap-shear samples prior to testing are shown in Figures 36-45. Note the extent of debonding and deformation observed in the bonded Marseal M-3500 samples after immersion and prior to testing. Lap-shear samples for the Marseal M-3500 material were cut from sections with as little as debonding as possible. In some cases, debonding had already occurred on the lap-shear samples at the far edge.

The exact cause for this behavior is unknown, but possibly related to both thermal and mechanical factors during immersion. Samples were only immersed at one temperature so possible variation in behavior at different temperatures is unknown. The immersion temperature (68°C) is slightly higher than the maximum use temperature for the Marseal M-3500 material (65°C). Variation in liner grain/reinforcement orientation may be a factor but samples were only tested in the as-received condition. Variation in thermal expansion and other chemical/mechanical characteristics between the Marseal M-3500 and Marseal 2000 materials may have also contributed to the behavior observed in those samples.

The bond length (not sample width) is not an identified parameter in the lap-shear test per ASTM D6392 but the bond length was approximately measured in case of significant lap-shear strength values in the test. As can be seen in Figures 46-53, both liner samples broke in the base material rather than peeling at the lap joint. This is a typical failure mode for samples with good adhesion (in absence of significant embrittlement), as the bonded edge provides a point of stress concentration.

Direct comparisons between the liner materials are difficult to make, as the bond joints are significantly different. The REMA 4CN material was joined using an angle splice joint with minimal overlap, while the Marseal M-3500 material was bonded using a true lap joint with significant overlap (~2.75”). The overlap amount did vary slightly among the samples but not significantly. Details of the bonding methods for both liner materials were not provided to SRNL/MS&T.

In contrast, the REMA 4CN material did not exhibit any notable debonding, permanent deformation or any other signs of degradation or variation from baseline condition, with the exception of surface precipitation. The REMA 4 CN liner joints were more consistent and did not show evidence of deformation or warping due to the immersion exposure. This behavior is more desirable from a liner integrity standpoint. It is unknown whether the Marseal M-3500 liner joint behavior would result in leakage in a typical installation, particularly if actual service temperatures are reduced. However, the debonding observed in these tests is certainly undesirable for a liner.

3.0 TEST RESULTS

Tensile and lap-shear data for the REMA 4CN and Marseal 3500 liner materials are shown in Tables 5 and Table 6. Elongation at break values were only obtained for the REMA 4CN material due to crosshead travel sensor failure on the Instron tensile machine. The elongation values for all REMA 4CN tensile samples were well beyond the minimum 370% reported on the Chemoline® 4CN data sheet, with no significant trend noted between baseline and immersed samples. The lowest elongation value recorded was 469% and that was for a baseline retest sample. All other values ranged from 845 to 1051%. It was observed during testing that elongation values for the Marseal M-3500 material were notably less than that of the REMA 4CN samples. However, variation in elongation values for baseline and immersed M-3500 samples was not readily distinguished. The Instron test machine has since been repaired, allowing elongation values for the Marseal 3500 material to be determined and reported separately if desired.

Table 5. Tensile Strength Data for Rema 4CN and Marseal M-3500 (units: psi)

	REMA 4CN Chemoline (#4 sheet)			**Marseal M-3500**		
Sample #	Baseline (#4)	50% S1	100% S1	Baseline	50% S1	100% S1
1	608.27	538.85	573.87	789.6	563.8	792.4
2	510.64	586.11	589.79	793.0	694.6	787.5
3	634.33	521.47	564.79	770.4	767.8	763.5
4	576.04	558.69	548.31	841.6	687.6	787.3
5	594.29	574.54	590.61	840.7	784.7	748.8
6				772.4	717.2	762.1
Average	584.71	555.9	579.76	801.28	702.62	773.6

Table 6. Lap-Shear Data for Rema 4CN and Marseal M-3500 liner materials (units: lb-force/inch)

	REMA 4CN Chemoline (#4 sheet)			**Marseal M-3500**		
Sample #	Baseline	50%	100%	Baseline	50%	100%
1	61.825	58.846	58.088	68.265	61.166	70.299
2	63.050	62.154	55.334	59.850	67.755	70.701
3	62.942	65.142	58.180	67.267	65.992	75.200
4	59.423	62.585	57.568	64.638	65.880	71.783
5	60.484	60.147	52.859	62.629	67.264	68.122
6				69.203		68.634
Average	61.5	61.8	56.4	65.309	65.611	70.789

No significant variations in tensile strength was observed for the REMA 4CN material, though the values for the 50% solution were relatively lower than either the baseline or 100% solution samples. The same trend is also observed for the Marseal M-3500 material and more pronounced. Based on the limited number of samples and the thin cross-section tested (Die D), it is difficult to say if this is a real trend of material degradation or an artifact of testing.

For the lap-shear tests, the REMA 4CN material showed lower strength values for the 100% solution than for either baseline or 50% solution samples. For the Marseal M-3500 samples, the opposite was observed, with the breaking load being higher for the 100% solution than for either baseline or 50% solution samples. Again, based on the limited number of samples tested, it is difficult to say if this is a real trend. In the case of the lap-shear tests, the bonds did not fail, with samples tearing apart essentially at the base material/lap joint interface. Samples were measured to see if these trends could be correlated to bond width variation, but no significant width variation was noted.

The measured tensile strength values of both materials were lower than the reported values by the manufacturers. The measured tensile strength values for the REMA 4CN material were approximately 80% of reported values ($\geq$ 725 psi), while the measured values for the Marseal M-3500 material were approximately 50% of the reported values. The reason(s) for these variations are unknown. Possible causes include ASTM die dimensions for measured vs. reported values, grain orientation (particularly for the Marseal M-3500 material), strain rate and other factors. A wider cross-section may be less sensitive to surface defects due to cutting or stamping methods, resulting in higher tensile strength values. ASTM D412 allows different die dimensions and strain rates to be used. The loads involved for the tensile samples were quite low due to the small sample cross-section, around 10 lbs or about 5% of the 200 lb load cell capacity. The loads required to tear the 1" wide lap-shear samples were much higher (~55-70 lbs). Since the purpose of these tests was to compare the results for baseline vs. immersed samples, rather than product qualification, these variations are not considered significant.

Durometer hardness testing was performed per ASTM D2240 using an A-scale indentor, Table 7 [41]. For the REMA 4CN material, the hardness was tested on the black side, as this side will presumably be exposed to fluid in service. The underlying gray side may influence overall hardness value but not significantly due to the thickness of the material. A few Durometer measurements were taken on the gray side for comparison, with the hardness values reading similar to the black side for the baseline sample, but for the immersed samples, the gray layer exhibited slight softening (~45-50A). This is not considered significant as the gray layer is presumed to be on the adhesive/concrete side and will not be exposed directly to the chemistry. The hardness of the REMA 4CN material is noted as 50+/-5A on the Chemoline data sheet [29]. The hardness of the Marseal M-3500 material is specified as 85+/-5A [30].

Given that a 5 point tolerance on Durometer hardness testing is typical, the variation observed for both materials is not significant. The hardness variation for the REMA material is minimal, with no obvious trend (average for all REMA samples is within 3.4 points). Hardness variation appears more significant for the Marseal M-3500 material, with the average hardness values being 4.2 and 6.2 points higher for the 50% S1 and 100% S1 samples, respectively. These results may suggest an increased hardness for the immersed samples and higher for the 100% solution, but variation in samples cut from different sheets cured at different times may also affect this behavior. All hardness values for the Marseal M-3500 samples are still within the 10 point tolerance typically quoted for Durometer hardness, with the exception of one reading.

Table 7. Durometer Hardness Data (Shore A scale)

Material/Condition	Reading 1	Reading 2	Reading 3	Reading 4	Reading 5	Average
REMA baseline	54	56	55	58	58	56.2
REMA 50% S1	50	52	52	55	55	52.8
REMA 100% S1	55	55	52	54	54	54.0
Marseal M-3500 base	82	82	83	80	82	81.8
Marseal M-3500 50%	85	86	87	88	84	86.0
Marseal M-3500 100%	90	88	86	91	85	88.0

4.0 DISCUSSION & OBSERVATIONS

Overall, both liner materials (REMA Chemoline 4CN and Marseal M-3500) are reasonably resistant to the S1 Saltstone bleedwater simulant, retaining significant original properties after immersion for 1000 hours at 68°C. Longer exposures may show more severe property changes, as thermo-oxidation and chemical effects may be more cumulative than shown in these tests. The Marseal M-3500 material appeared to show slight increase in hardness for the immersed samples, though the variation is still within the tolerance range for baseline values.

A qualitative observation on the lap-shear samples (post-testing) is that the Marseal M-3500 samples could be torn apart by hand with some effort, progressively easier for baseline, 50% solution and 100% solution samples, respectively. The baseline Marseal M-3500 sample required significant twisting and force, but could still be torn apart. In comparison, none of the REMA 4CN materials could be torn apart by hand using similar effort, with no noticeable difference between baseline, 50% solution and 100% solution samples. Variation in product thickness may affect this to some degree and the significance of this behavior for in-service performance is unknown, but this is likely due to the inherent difference between a true thermosetting (crosslinked) bromobutyl elastomer and a modified thermoplastic or copolymer.

While both materials showed reasonable lap-shear bond strengths, the REMA 4CN material exhibited superior bond quality and consistency. It is assumed that the materials were bonded per manufacturer instructions, though SRNL/MS&T did not confirm this aspect. The Marseal M-3500 material exhibited delamination or “puckering” during simulant immersion. The exact cause and temperature at which the delamination behavior initiates is unknown. The behavior is likely related to the thermoplastic or modified thermoplastic nature of the Marseal M-3500 liner based on PVC and EVA polymers as compared to the true thermosetting and elastomeric nature of the bromobutyl rubber. It is also noted that the maximum service temperature of the Marseal M-3500 material is stated as 150 °F (65 °C) by the material manufacturer. The basis for the temperature limit is unknown, but it is slightly lower than the 68 °C immersion test temperature. Lower temperatures may or may not cause similar behavior, or the behavior may simply take longer to manifest.

Another aspect of the Marseal M-3500 material to consider is the presence of polyester reinforcement. Polyesters can be sensitive to hydrolysis and degradation by high pH solutions, particularly at elevated temperature. The Marseal M-3500 material is rated as suitable for high pH solutions by the manufacturer, but additional testing may be needed to evaluate possible long-term effects on the reinforcement. A similar concern was noted in Reference 42, in which a similar material (XR-5 8130, EIA, blend of PVC and Elvaloy®) was evaluated along with several other polymers including HDPE. The XR-5 geomembrane is commonly used in wastewater and landfill liner/containment applications [18].

In Reference 42, candidate liner materials were immersed in a simulated DSSF (double shell slurry feed) solution to evaluate resistance to leachate or bleedwater from low-level liquid radioactive waste at Hanford proposed to be solidified in a cement-based grout matrix, similar to Saltstone, in the Hanford Grout Disposal Program (HGDP) [42]. The immersion temperature used was 90 °C, which is much higher than 68 °C used in the current SDU6 liner studies. The pH of the DSSF was not given but the solution was noted to contain 2M hydroxide. Immersion tests were 120 days, and were needed to address higher steady-state temperatures (90 °C) in the Hanford vault than previously identified (75 °C). The report notes that the liner should not see actual fluid but immersion tests were performed as a worst-case scenario. Later design changes placed the liner on the outside of the vault and the HGDP program was later cancelled by the DOE.

Reference 42 also included exposure to some organics in the DSSF simulant, but the organics included were edetates (added as tetra-sodium edetate trihydrate, 3.53 g/L) and citrates (added as tri-sodium citrate dihydrate, 2.06 g/L). The authors of Reference 42 correctly note that organics are a principal cause of polymer degradation. However, EDTA and citric acid are not known to attack any of the materials that were tested.

Liner materials were gamma-irradiated to cumulative doses of approximately 0.6 Mrad, 3.6 Mrad, 16 Mrad, and 39 Mrad. These levels were reportedly representative of Hanford Grout Disposal vault liner dose equivalents of 120 days, 2 years, 10 years, and 30 years. Comparatively, the radiation dose anticipated for the SDU6 liner is much lower over 25 years (0.82 Mrad, Reference 47). Reference 42 did not address dose rate effects for the materials.

Conclusions of Reference 42 were that HDPE was judged to be chemically compatible with DSSF at 90 °C, with the only properties of concern being liner puncture force and the liner puncture elongation attributed to thermal degradation. A polypropylene liner was a1so judged chemically compatible with DSSF at 90 °C.

The other three liners (nonreinforced Hytrel® polyester, polyurethane and XR-5 PVC/Elvaloy®) were deemed incompatible with DSSF at 90 °C, with testing discontinued at various times for each material. The authors suspected that the primary cause of each liner failure was a temperature-induced hydrolysis of esters (or ester components). The effect of radiation on liner compatibility was expected to be negligible for the application, as significant property decreases (elongation) were only significant at doses ≥16 Mrad and design changes to the vault would significantly decrease the radiation dose to the liner.

Direct comparison between the liner materials and performance in the Hanford grout liner study and the SDU6 testing cannot be made due to variation in materials, simulant chemistry and immersion temperature. In particular, the 90 °C immersion temperature is significantly higher than anticipated in the SDU6 application and above the maximum service temperatures of both 4CN and M-3500 materials. However, inclusion of organics and radiation exposure as in the Hanford liner study is worth considering in future SDU liner testing. Longer immersion periods (120 days in the Hanford study vs. 1000 hours in the SDU6 liner study) were also noted. Specific comparison between the XR-5 ethylene-interpolymer alloy (EIA) based on PVC/Elvaloy® and the Marseal® M-3500 material is not known at this time, but the materials are similarly described with similar properties. Unfortunately, the Hanford grout liner study did not include a butyl or halobutyl elastomer liner for comparison.

A literature review of butyl rubber linings in contact with alkaline solutions combined with ionizing radiation was not performed. Such a review may be prudent to provide additional technical basis for selection and use of the REMA 4CN material in the SDU6 application. The available data in the literature on such linings in such environments is likely limited. At least one reference was found for butyl rubber seals evaluated after exposure to alkaline Hanford Tank waste simulant at three temperatures (18, 50, 60 °C) for periods up to 180 days. Samples were also exposed to radiation doses of 143, 286, 571 and 3,670 krad (3.67 Mrad). Dose rate effects were not evaluated. The butyl rubber seals showed satisfactory results, though less satisfactory than EPDM [43].

The pre-immersed samples were presumably prepared as they would be during actual installation prior to service. If the Marseal M-3500 samples were incorrectly prepared or bonded, additional testing may be warranted to further evaluate this aspect. It is unclear if this behavior would absolutely result in leakage in a given liner configuration or scenario, but the observed delamination is undesirable, particularly if the liner is solely credited for leaktightness for the SDU design. It is also noted that none of these samples were subjected to 41 foot head pressure, so joint integrity of either material at bounding hydrotest conditions cannot be directly determined from this evaluation.

Delamination of the Marseal M-3500 material from the epoxy-coated concrete blocks was less severe than for the polymer-to-polymer bonded samples but was still observed, particularly at corners, even without imposed stress (blunt screwdriver). At these locations, the material could be pulled or peeled away from the substrate without significant effort. This is a different configuration than for the lap-shear bonded or seamed samples. Therefore, the overall bonding quality of the REMA 4CN material is superior to that of the Marseal M-3500 material, at least under the conditions observed.

The liner will be loaded by the grout after operations begin. However, if the liner has any tendency to locally debond due to thermal or mechanical stress, this could provide a leak path for contaminated bleedwater. In the Marseal 3500 samples tested, the debonded area was limited and did not extend the full bond length of the lap joint. In addition, the lap-shear samples failed in the base material rather than peeling apart, indicating overall good adhesion even with the observed delamination. However, this behavior raises concern and should be further evaluated prior to final selection or large-scale installation of the Marseal M-3500 material. This behavior may not manifest before or during hydrotesting, but only after exposure to elevated temperature in service.

The 1000-hour immersions and post-exposure tests were primarily performed to screen the two liner options for severe incompatibility and general behavior. Such exposures, though useful for direct comparison, are of limited value for service life prediction. Additional tests and analytical methods would have to be employed to provide a more detailed evaluation of potential service effects on the liner materials. Some additional testing such as weight/volume change, thermal-mechanical analysis (TGA, DSC, DMA), or compositional analysis (via FTIR spectroscopy) could be performed on the remaining samples. However, such data are still likely of limited value since the exposure time is so short relative to the intended service life. A 1000 hour exposure is only ~1.9% of a 6 year operating life, and only ~0.5% of a 25-year service life.

Longer-term real-time aging and/or accelerated-aging tests are therefore recommended to more comprehensively address liner service life. A combination of real-time aging and accelerated-aging tests is likely needed, as both methods have distinct advantages and drawbacks.

The main advantage of real-time aging at bounding conditions is that significant aging data can often be obtained without significant expense. Real-time aging data are always desired since the aging conditions represent actual service, using aging times as long as practical. Using a simple 10% rule-of-thumb, immersion tests of 6 months to 2.5 years would be needed to address a 6 to 25 year service life. Longer aging periods could be performed, more as a surveillance program with limited sampling and testing.

The primary disadvantage of real-time aging is that extensive test periods are often needed before any meaningful property changes are observed, limiting predictive value. Accelerated-aging methodologies (Arrhenius, Eyring, etc.) are therefore often used to more aggressively challenge the materials within more reasonable aging periods, providing more predictive value than real-time aging methods.

For accelerating-aging, the property or parameter used to evaluate material performance should be the most relevant for service. Using several parameters may be preferable, unless the single parameter selected is clearly the most important for performance. For a bonded tank lining primarily subject to hydrostatic or compressive loads, the most relevant failure mechanism is likely either embrittlement of the liner itself (elongation %) or bond integrity loss (due to combination of stress, hydrolysis effects, heat/radiation effects). The specific failure point is unknown, but significant embrittlement is likely needed before failure occurs.

For polymeric materials exposed to multivariable environments (heat, radiation, oxidation, chemicals, stress), several properties and analytical techniques may be of interest. Elongation, tensile strength, weight/volume change, density, permeability, degree of oxidation (carbonyl index, oxidation-induction time (OIT)) and other properties may be of interest. For example, OIT testing via DSC (differential scanning calorimetry) is often used to evaluate HDPE geomembranes and pressure piping for resistance to thermo-oxidation and antioxidant stability [12].

A concern with accelerated-aging is if overly aggressive aging conditions are used, the degradation mechanism may vary from that which occurs in actual service, such that the failure time at actual service conditions may be inaccurately predicted. Nonlinear aging mechanisms or non-Arrhenius aging behavior can complicate life prediction. Antioxidant consumption, diffusion-limited oxidation, crosslinking vs. scission processes and other mechanism variations can influence polymeric material behavior. Therefore, care must be taken to select the accelerated-aging scheme.

ASTM standards such as ASTM D3045, Standard Practice for Heat Aging of Plastics Without Load, require a minimum of four aging temperatures to be used, with the lowest aging temperature being 10-20 °C more than the service temperature [44]. Ideally, accelerated-aging causes at least one critical property or performance parameter to reach a failure point over time at a range of conditions, facilitating predictive methodology such as time-temperature superposition (TTS).

The accelerated-aging scheme might also affect one material more severely than others. As an example, the maximum use temperature of the Marseal M-3500 material is quoted as 65 °C, whereas the upper temperature limit for the Chemoline 4CN material is 85 °C. Higher aging temperatures would likely be needed to challenge the Chemoline 4CN material than the Marseal M-3500 material.

Similarly, radiation dose and dose rate effects may influence liner material behavior. The cumulative dose expected for the SDU6 liner (1 Mrad over 25 years) is not highly challenging for most polymeric materials. However, butyl rubber is relatively sensitive to ionizing radiation compared to other elastomers and the cumulative dose is not completely benign, particularly if dose rate or synergistic effects are significant. Many polymers are known to be sensitive to the dose rate, though such effects are more pronounced in oxygen-bearing or air environments. Inert or low-oxygen environments tend to mitigate dose rate effects [45-46]. The effect of oxidation and dose rate to a bonded liner sandwiched between concrete and Saltstone grout is unknown, but likely less severe than for in-air service.

The radiation resistance of bromobutyl rubber for the SDU6 liner application was briefly addressed in Reference 47. This document provides an estimate of the dose to the liner over the service life and provides some information on the radiation tolerance of butyl rubber and halobutyl derivatives. This document acknowledges that synergistic and dose rate effects may influence liner stability, though significant effects are not anticipated at the estimated cumulative dose. It is noted that in the Hanford grout vault liner study (Reference 42), radiation was imposed on candidate liner materials and was not considered to cause significant degradation until doses >16 Mrad were reached. However, butyl rubber was not included in that study and dose rate sensitivity was not evaluated.

The dose rate sensitivity of a given polymer can only be evaluated by irradiating the material at varying dose rates and comparing specific properties. A limitation is that unless the dose rate imposed in testing is equivalent to the service dose rate, it can only be assumed that the degradation mechanism is the same for both dose rates. Another limitation is that at lower dose rates, long irradiation times may be needed to cause meaningful property changes. A dose rate study of 1 year is typical, though different periods can be used.

For example, a 25-year cumulative dose of ~1 Mrad dose could be imposed at 115-150 rad/hr (~9-12 months), 300-500 rad/hr (~3-6 months), 1500 rad/hr (~1 month), and 2E+05 rad/hr (max dose rate, 5 hours) or similar. In that case, the lowest dose rate (115 rad/hr) would be ~25X the actual average dose rate of 4.5 rad/hr. Even if failure points are not reached, the variation in response for different dose rates could be evaluated.

Another approach is to irradiate material at maximum dose rates to various doses until significant damage or failure parameters are reached. The advantages of this approach are that data can be obtained relatively quickly and the dose to failure for the given parameter(s) of interest can be determined. The larger the margin between the dose to failure and the cumulative service dose, the less likely the service dose will cause significant degradation. However, this approach alone should not be used to evaluate service life.

Service life evaluation of the SDU6 liner is recommended not only for SDU6 performance assessments or other design/safety basis documentation, but also for future SDU designs and other SRS applications involving similar service conditions. Comparable aging data for specific materials exposed to particular service conditions are often difficult if not impossible to find, limiting direct comparisons. A particular limitation of the SDU application is that interior linings or coatings cannot be inspected or examined after installation and grout placement, so obtaining service-aged data is not possible.

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Both liner materials (REMA Chemoline® 4CN and Marseal® M-3500) retained significant tensile properties after immersion for 1000 hours in the S1 Saltstone bleedwater simulant at 68 °C. Slight hardness increase was observed for the Marseal M-3500 material with immersion, though within baseline hardness tolerances. Longer exposures may show more severe property changes, as thermo-oxidation and chemical effects may be more cumulative.

5.2 The REMA 4CN material exhibited superior resiliency and bond quality compared to the Marseal® M-3500 material at the imposed test conditions. Warping and debonding of the Marseal® M-3500 samples was observed. Based on these observations alone, the REMA 4CN material is a preferred liner. The specific cause of the Marseal M-3500 debonding behavior and the threshold time/temperature values at which it occurs is unknown. The immersion temperature used in these tests (68 °C) is slightly higher than the service temperature limit of the Marseal M-3500 material (65 °C). The basis for the limits is not clearly defined but the service temperature limit of the REMA 4CN material (85 °C) is much higher. The behavior may also be attributed to the inherent differences in the polymers. The REMA 4CN liner is a true thermosetting elastomer, whereas the Marseal® M-3500 liner is a modified thermoplastic or thermoplastic elastomer blend.

5.3 The tests performed did not involve exposing liner materials to ionizing radiation or organics that may exist in future Saltstone feed streams. The REMA 4CN bromobutyl rubber is likely more sensitive to ionizing radiation, while the Marseal® M-3500 material may be more sensitive to certain organics, if present. The estimated radiation dose absorbed by the liner in service is not severe (~0.82 Mrad over 25 years). However, synergistic and long-term effects of all environmental variables may occur. In addition, future SDU liners, if needed, may be exposed to higher dose rates.

5.4 Longer-term real-time aging and/or accelerated-aging tests are recommended to evaluate liner service life. Both approaches have limitations so a combined approach is usually best. Several analytical techniques and parameters can be used to evaluate liner performance. Such tests are not particularly difficult to perform and can provide longer-term performance data for future SDU designs and other SRS applications. Additional candidate liner materials may also be included.

5.5 Lower service temperatures, if applicable, will likely reduce thermal/mechanical stresses and possibly chemical and thermo-oxidation effects on the liner. Such temperatures should be incorporated into future SDU coatings and lining tests. Lower service temperatures may also broaden material selection options.

5.6 All polymeric coating/liner materials can offgas due to thermal and/or radiolytic mechanisms. The amount and species of gas evolved depends on several factors. The Marseal® M-3500 and 2000 PVC/EVA blends can possibly evolve hydrocarbon cases as well as HCl gas due to dehydrochlorination. Thermal dehydrochlorination will not likely occur below 80 °C. Threshold radiation doses for gas generation for the liner materials are unknown. Upon sufficient irradiation, the REMA 4CN bromobutyl rubber will likely evolve hydrogen and methane, with hydrogen bromide (HBr) and bromine also possibly evolved. The impact of liner gas generation may require additional evaluation.

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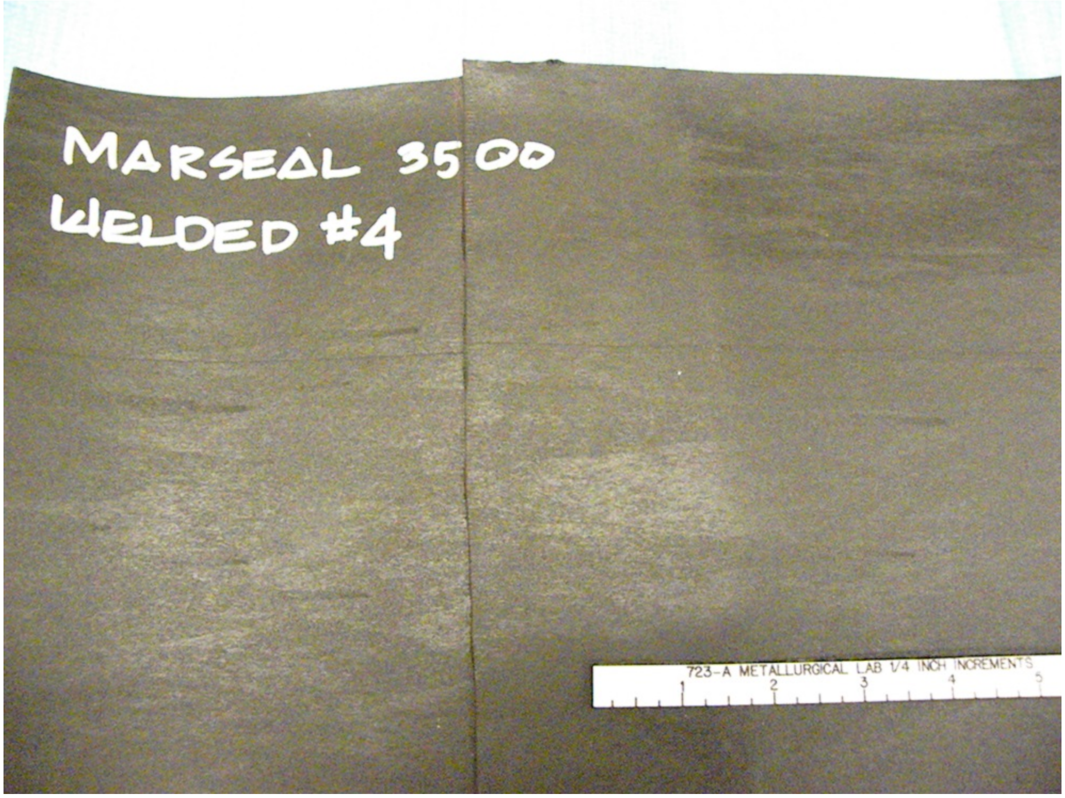

Figure 1. Marseal 3500 welded #4 – baseline (non-immersed)

Figure 2. Marseal 3500 welded #5 – baseline (non-immersed)

Figure 3. Marseal 3500 #4 – intact/baseline (non-immersed)

Figure 4. Marseal 3500 #5 – intact/baseline (non-immersed)

Figure 5. Marseal 3500 inside corners – baseline (mislabeled, Marseal® 2000)

Figure 6. Marseal 3500 outside corner – baseline (mislabeled, Marseal 2000)

Figure 7. Marseal 3500 outside corner#5 – baseline (mislabeled, Marseal 2000)

Figure 8. Marseal 3500 welded#5 – baseline lap-shear samples (pre-test)

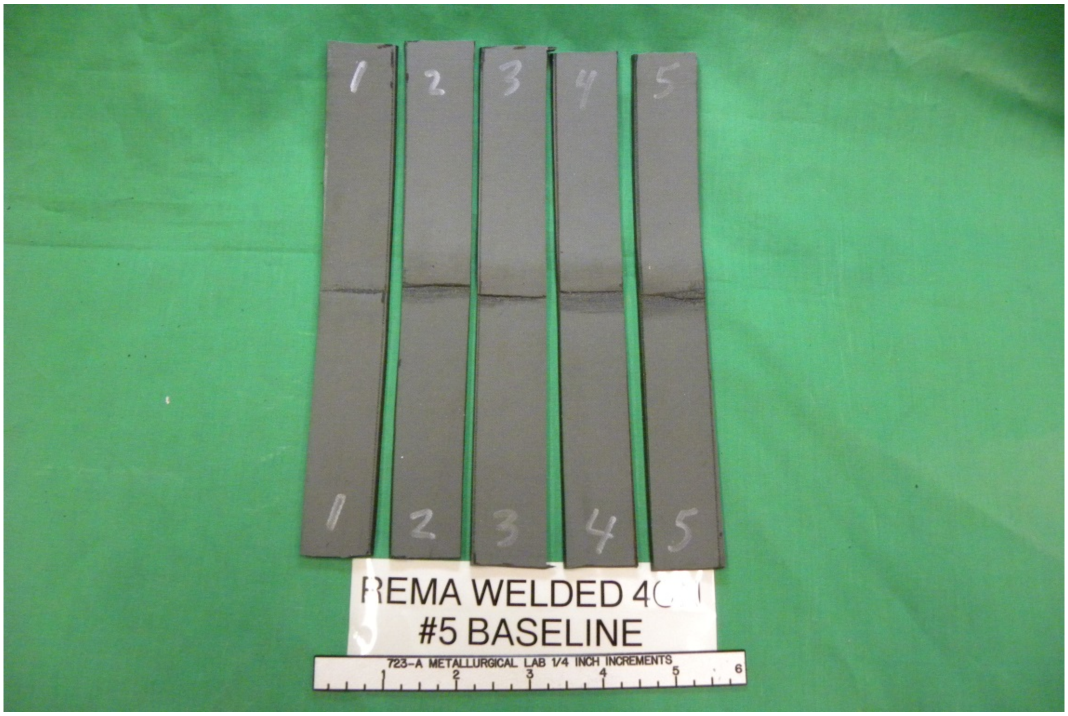

Figure 9. REMA 4CN welded#5 – lap-shear samples (pre-test)

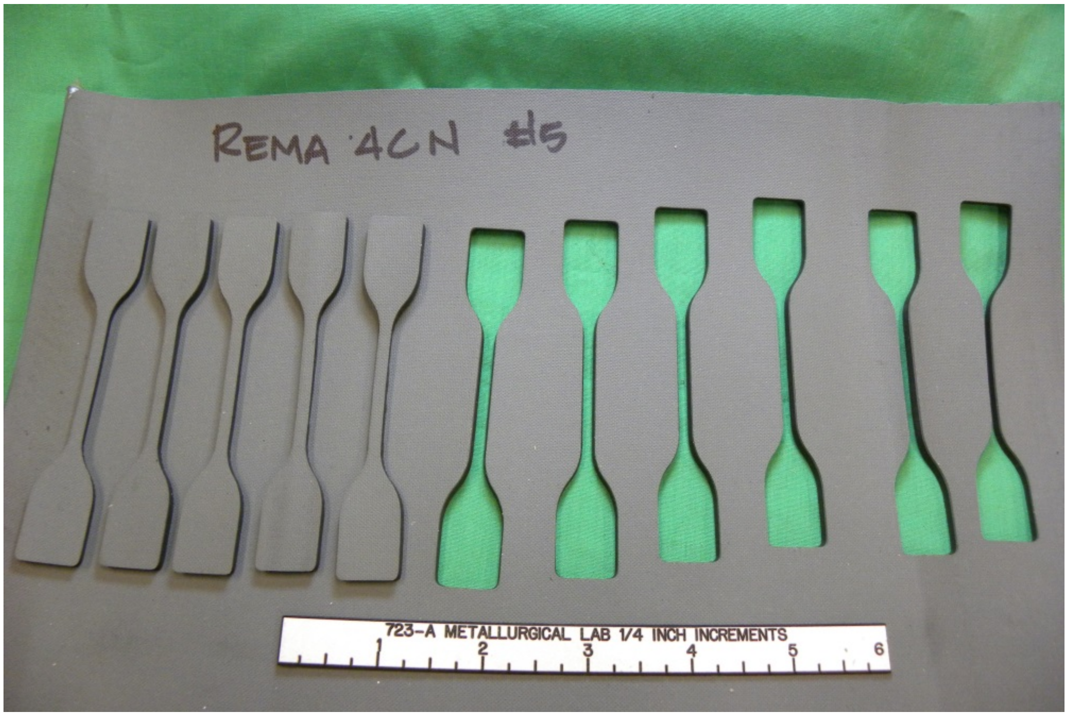

Figure 10. REMA 4CN baseline #5 – tensile samples (pre-test)

Figure 11. 1SREL-LS-MAR-100-052416 (Marseal 3500, bonded, 100% S1 solution)

Figure 12. 2SREL-LS-MAR-050-052416 (Marseal 3500, bonded, 50% S1 solution)

Figure 13. Post-immersion debonding of 2SREL-LS-MAR-050-052416
(Marseal 3500, bonded, 50% S1 solution)

Figure 14. 3SREL-LI-MAR-100-052416 (Marseal 3500, intact, 100% S1 solution)

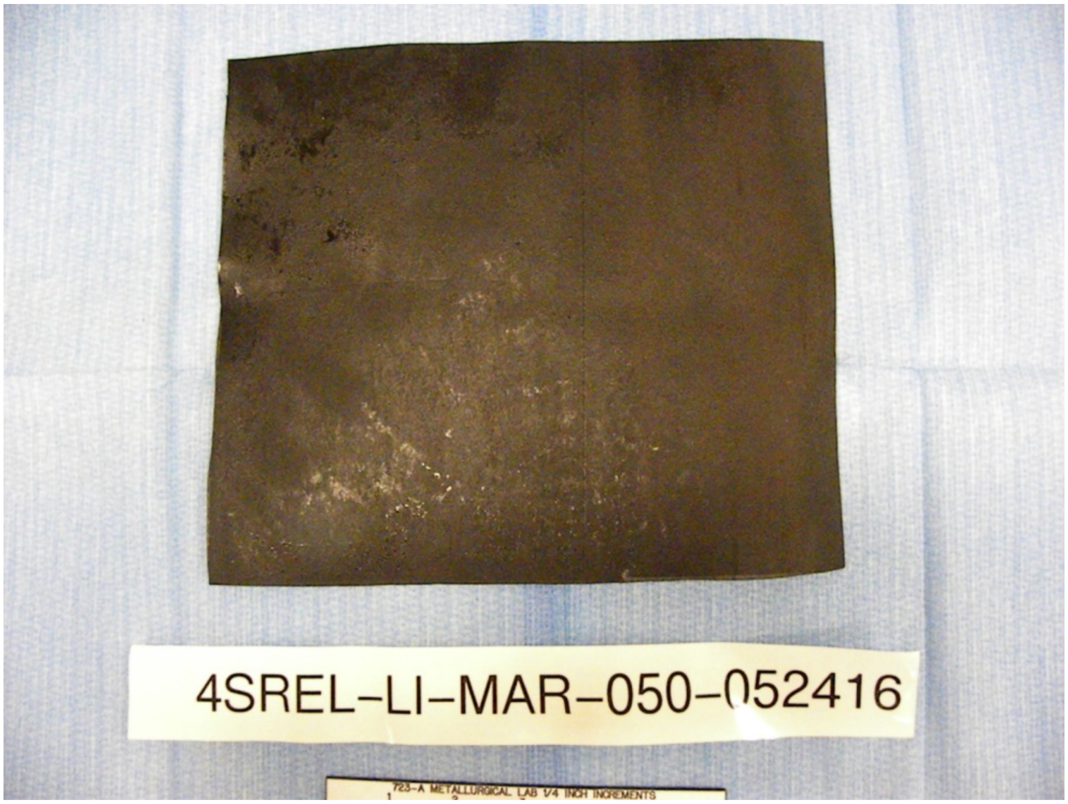

Figure 15. 4SREL-LI-MAR-050-052416 (Marseal 3500, intact, 50% S1 solution)

Figure 16. 9SREL-CI-MRU-050-052416 (Marseal 2000, corner/intact, 50% S1 solution)

Figure 17. 10SREL-CI-MRU-100-052416 (Marseal 2000, corner/intact, 100% S1 solution)

Figure 18. 11SREL-CS-MRU-050-052416 (Marseal 3500/2000, bonded, 50% S1 solution)

Figure 19. 11SREL-CS-MRU-050-052416 (opposite side of Figure 18.)

Figure 20. 12SREL-CS-MRU-100-052416 (Marseal 2000/M-3500, bonded, 100% S1 solution)

Figure 21. 12SREL-CS-MRU-100-052416 (opposite side from Figure 20)

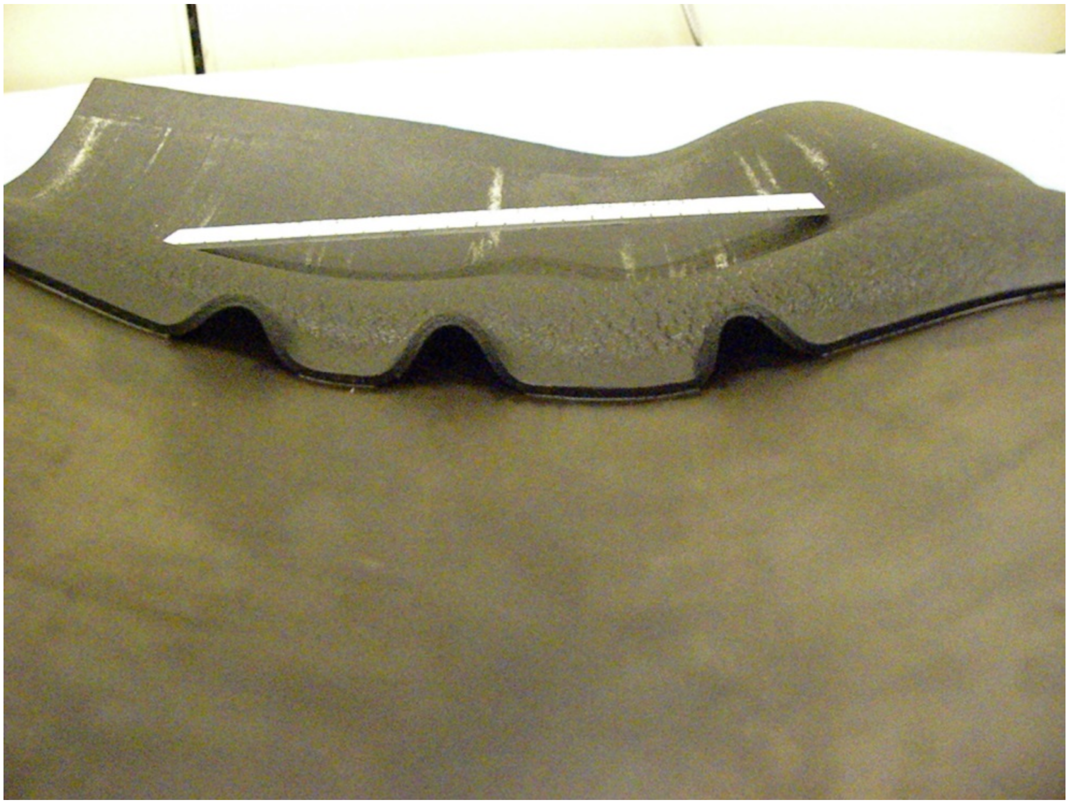

Figure 22. 12SREL-CS-MRU-100-052416 (note surface wrinkling and degree of warping)

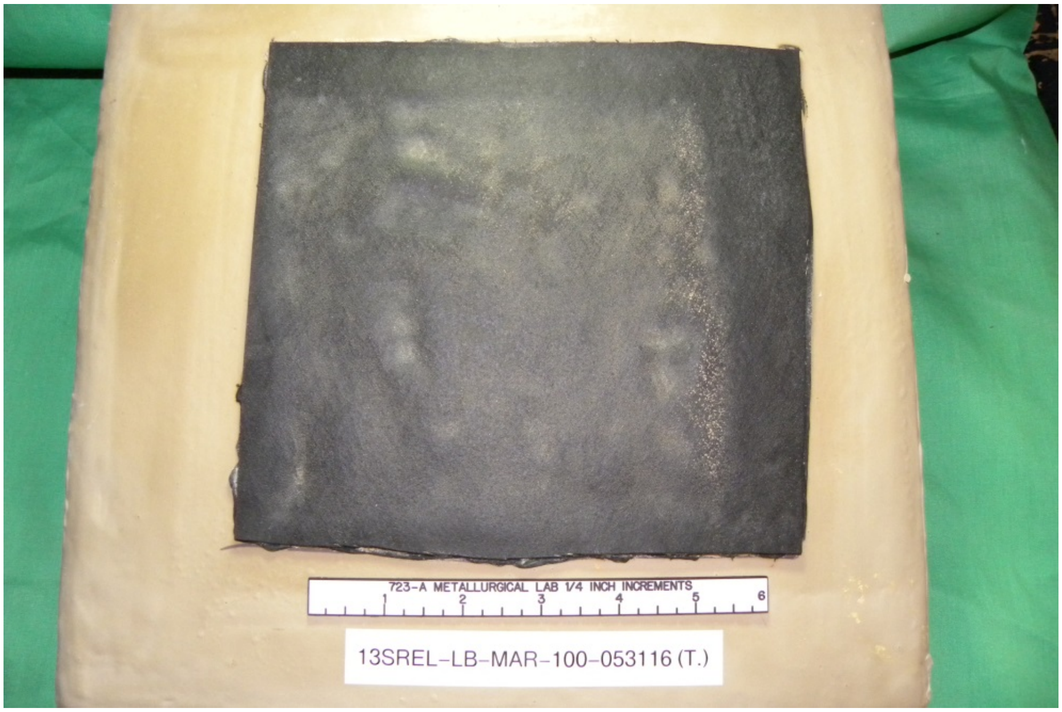

Figure 23. 13SREL-LB-MAR-100-053116
(Marseal M-3500 bonded to EC-66 epoxy/concrete, 100% S1 solution)

Figure 24. 13SREL-LB-MAR-100-053116
(slight delamination/debonding at corners, as-received, no probing)

Figure 25. 13SREL-LB-MAR-100-053116
(delamination/debonding at opposite corners, as-received, no probing)

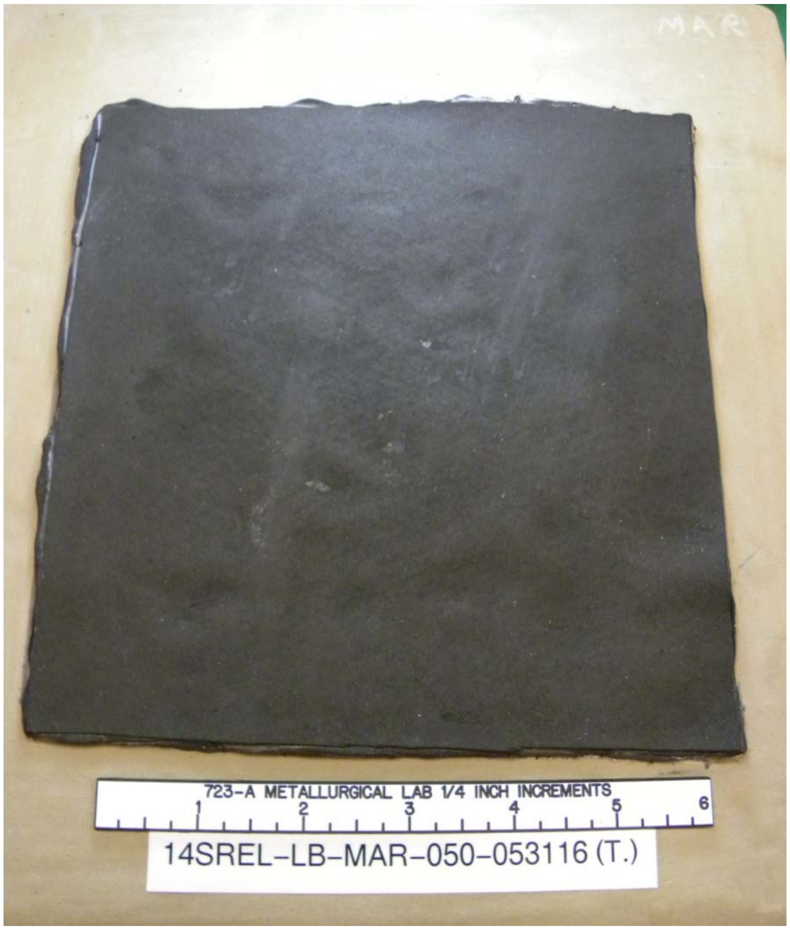

Figure 26. 14SREL-LB-MAR-050-053116
(Marseal M-3500 bonded to EC-66 epoxy/concrete, 50% S1 solution)

Figure 27. 14SREL-LB-MAR-050-053116 (note corner debonding, as-received, no probing)
(Marseal M-3500 bonded to EC-66 epoxy/concrete, 100% S1 solution)

Figure 28. 14SREL-LB-MAR-050-053116 (mid-width debonding, as-received, no probing)
(Marseal M-3500 bonded to EC-66 epoxy/concrete, 100% S1 solution)

Figure 29. 14SREL-LB-MAR-050-053116 (corner debonding, as-received, no probing)
(Marseal M-3500 bonded to EC-66 epoxy/concrete, 100% S1 solution)

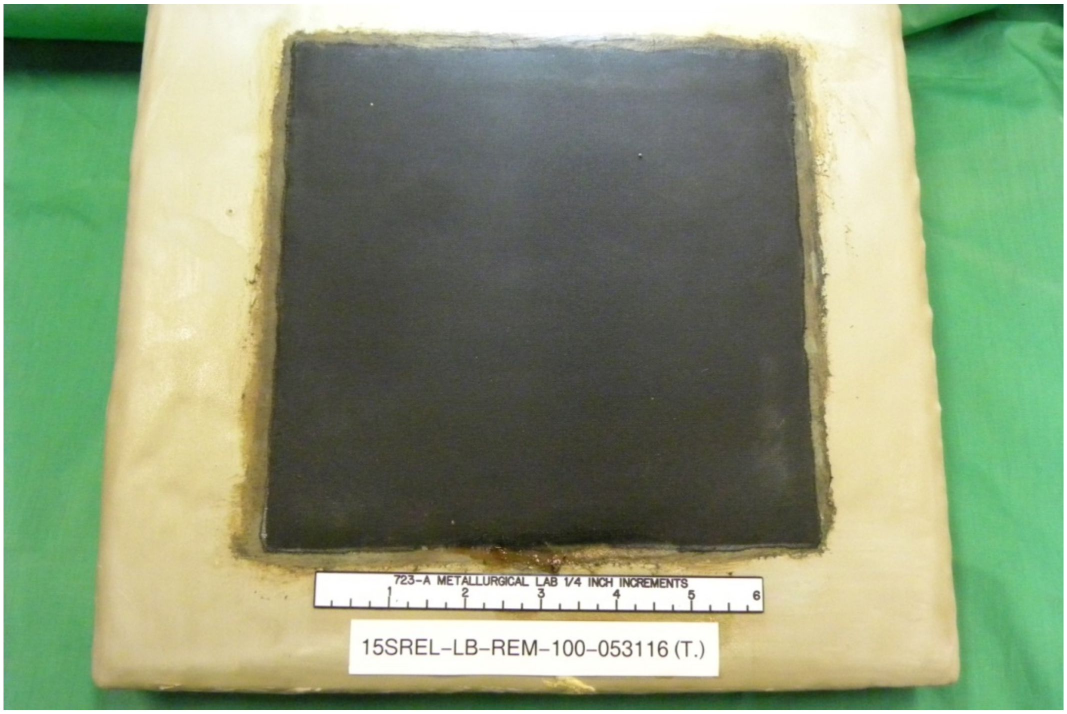

Figure 30. 15SREL-LB-REM-100-053116 (as-received, no probing)
(REMA 4CN bonded to EC-66 epoxy/concrete, 100% S1 solution)

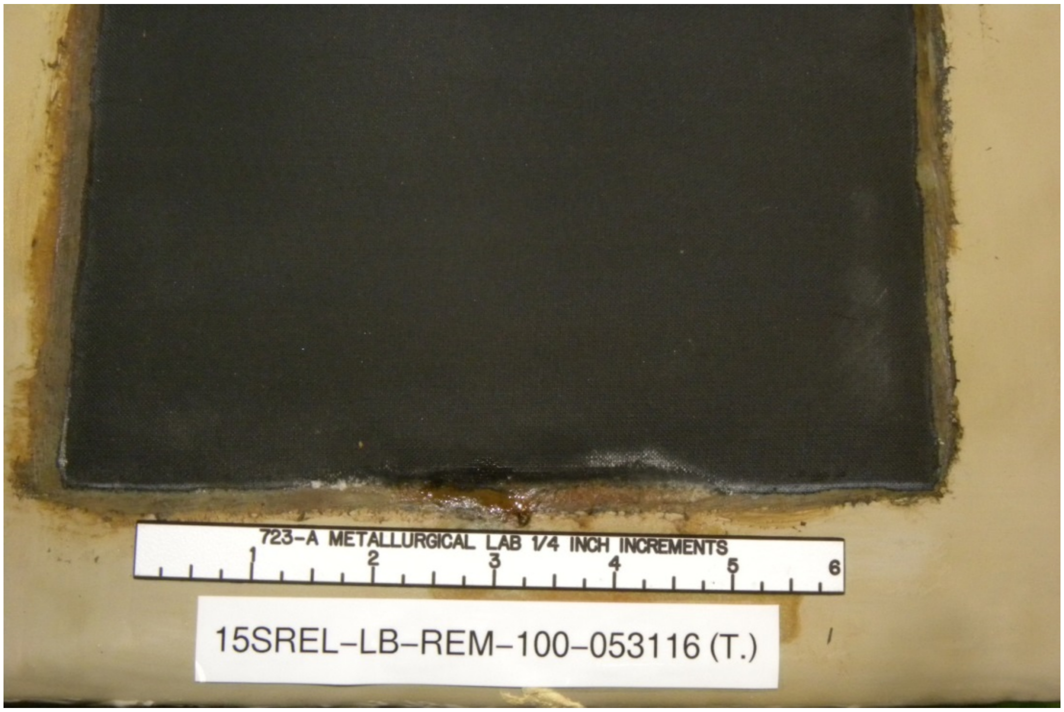

Figure 31. 15SREL-LB-REM-100-053116 (as-received, no probing)
(closer view, fluid seeping from liner edge after immersion)

Figure 32. 15SREL-LB-REM-100-053116 (no delamination observed, representative of all corners)

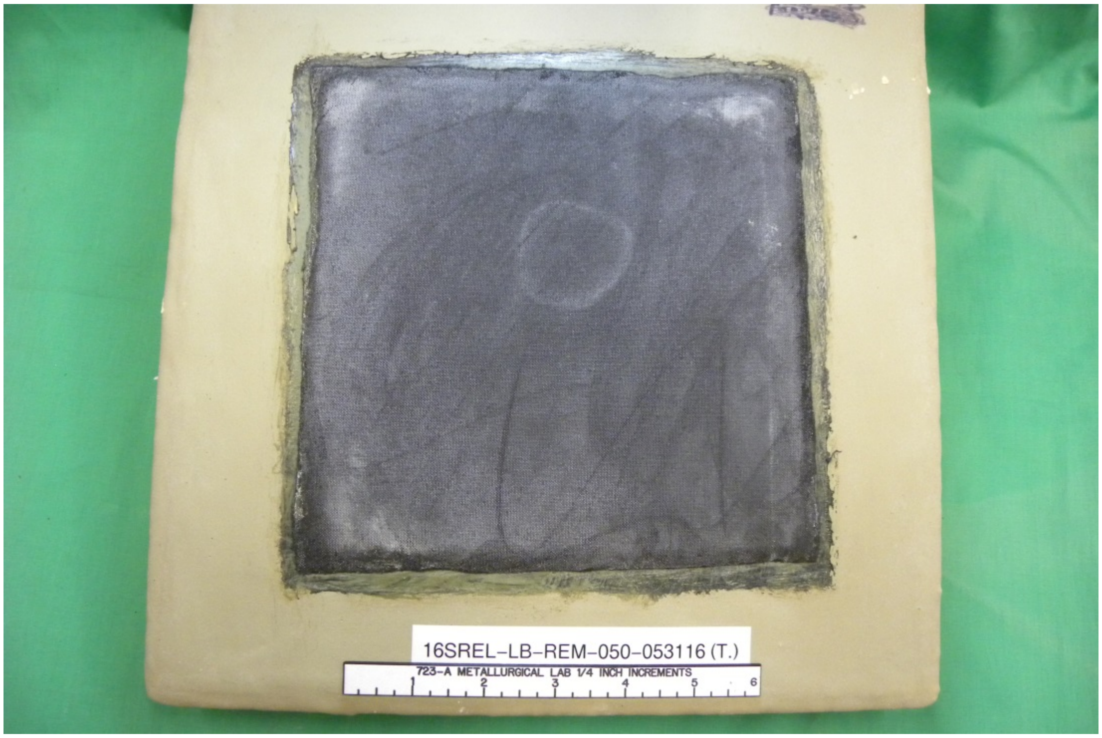

Figure 33. 16SREL-LB-REM-050-053116
(REMA 4CN bonded to EC-66 epoxy/concrete, immersed in 50% S1 solution)

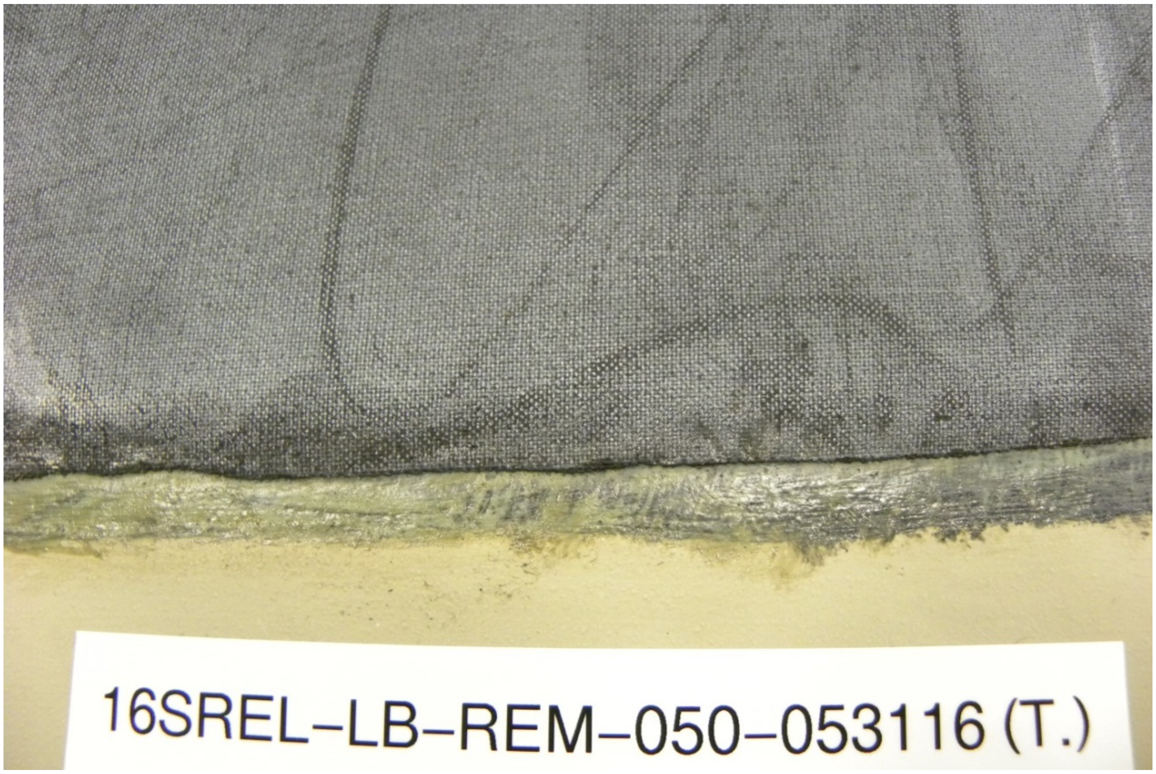

Figure 34. 16SREL-LB-REM-050-053116 (no delamination observed as-received, thinner cement layer)

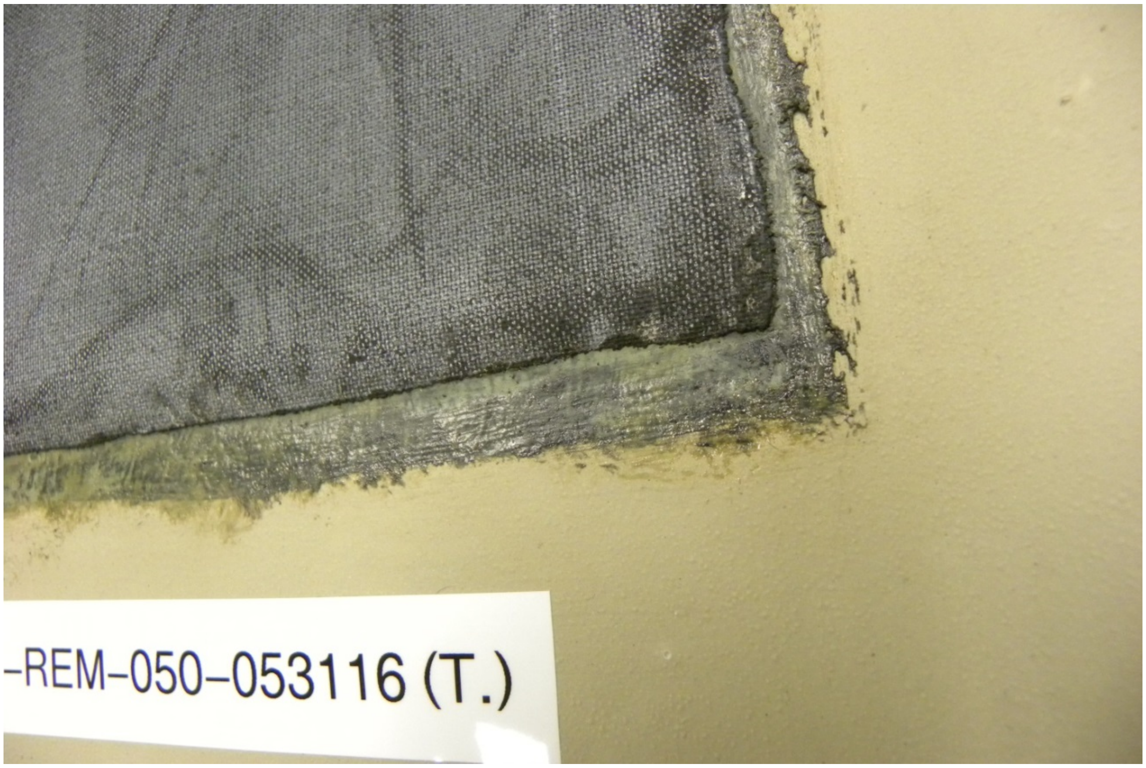

Figure 35. 16SREL-LB-REM-050-053116 (no delamination observed, representative of all corners)

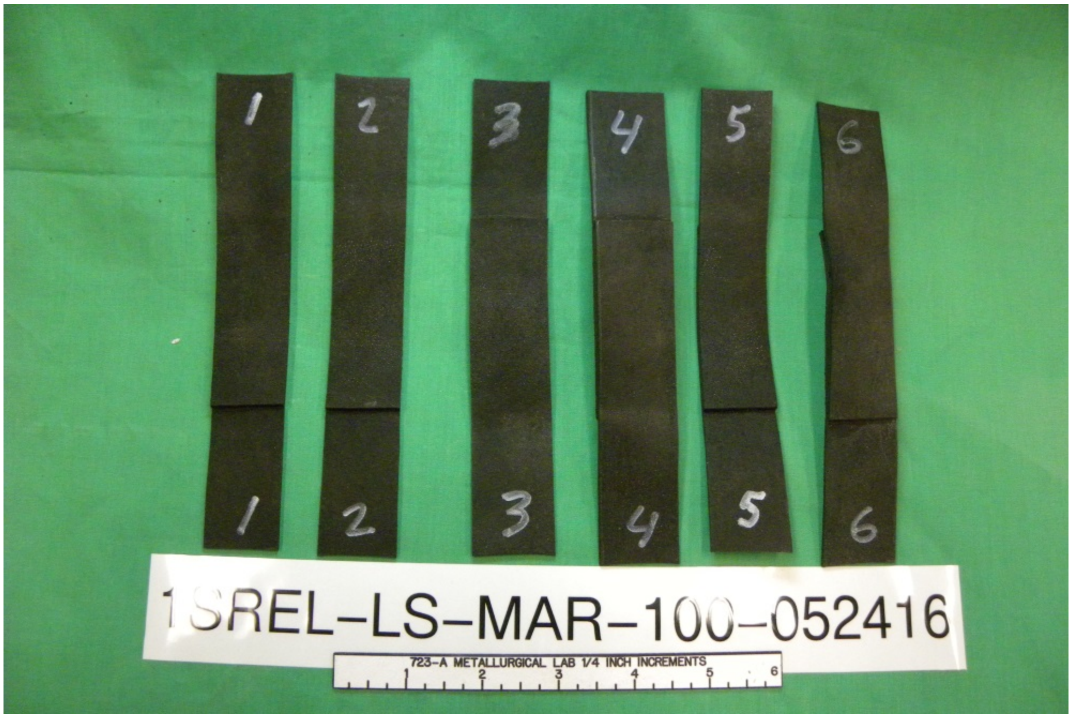

Figure 36. Marseal M-3500 welded– lap-shear samples (100% S1 solution) – pre-test

Figure 37. Marseal 3500 welded– lap-shear samples (100% S1 solution) – pre-test delamination

Figure 38. Marseal M-3500 welded– lap-shear samples (50% S1 solution) – pre-test

Figure 39. Marseal M-3500 welded– lap-shear sample#1 (50% S1 solution) – pre-test debonding

Figure 40. Marseal 3500/Marseal 2000 lap-shear samples (50% S1 solution) – pre-test

Figure 41. Marseal M-3500/Marseal 2000 lap-shear sample #4 (50% S1 solution) – pre-test debonding (note texture difference between Marseal M-3500 with polyester reinforcement and Marseal 2000

Figure 42. Marseal M-3500/Marseal 2000 lap-shear samples (100% S1 solution) – pre-test debonding

Figure 43. Marseal M-3500/Marseal 2000 lap-shear sample (100% S1 solution)
(note deformation/debonding – pre-test)

Figure 44. Marseal M-3500 lap-shear samples (50% S1 solution) – pre-test debonding
(approximately 2.75" overlap, debonded length approximately ½")

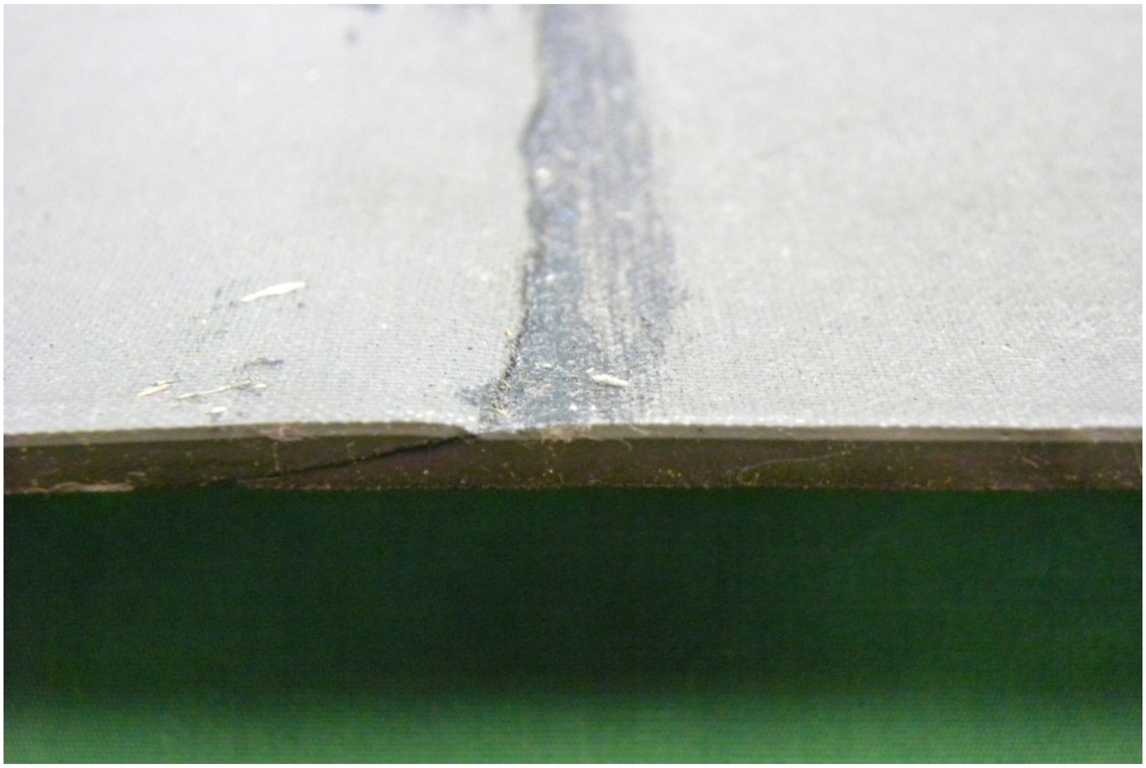

Figure 45. Typical splice joint configuration for REMA 4CN material

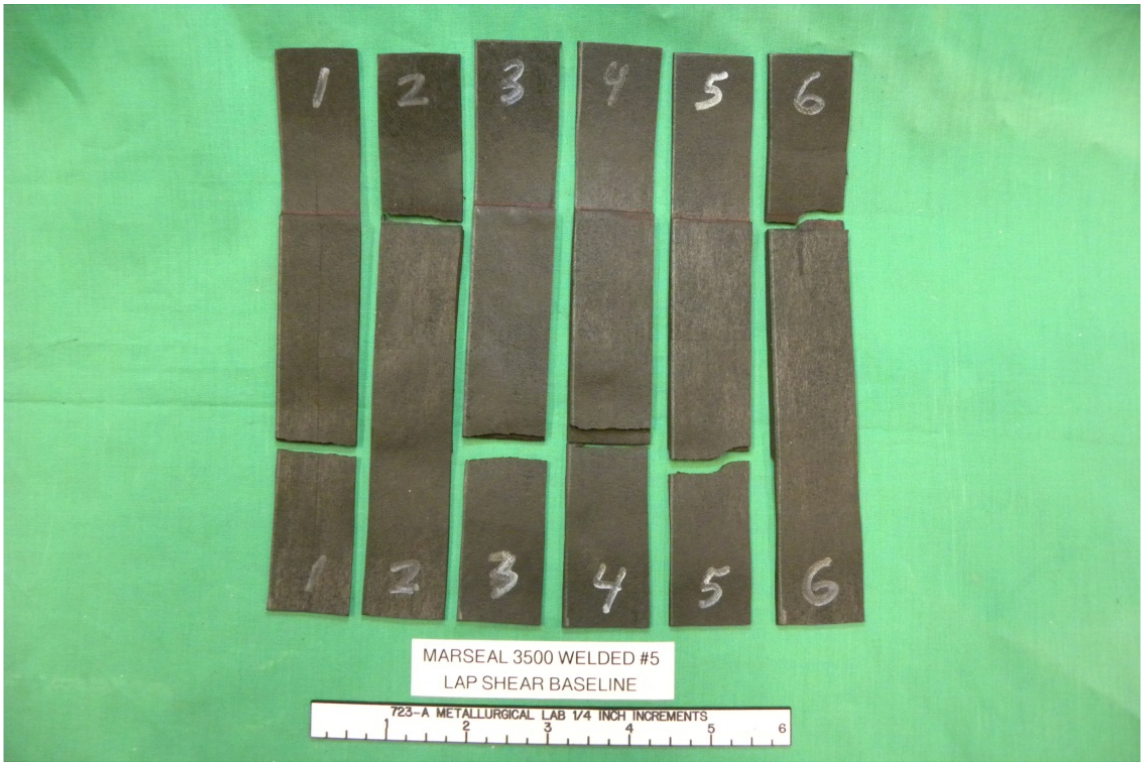

Figure 46. Marseal M-3500 lap-shear samples (baseline) – post-test (failure in base material)

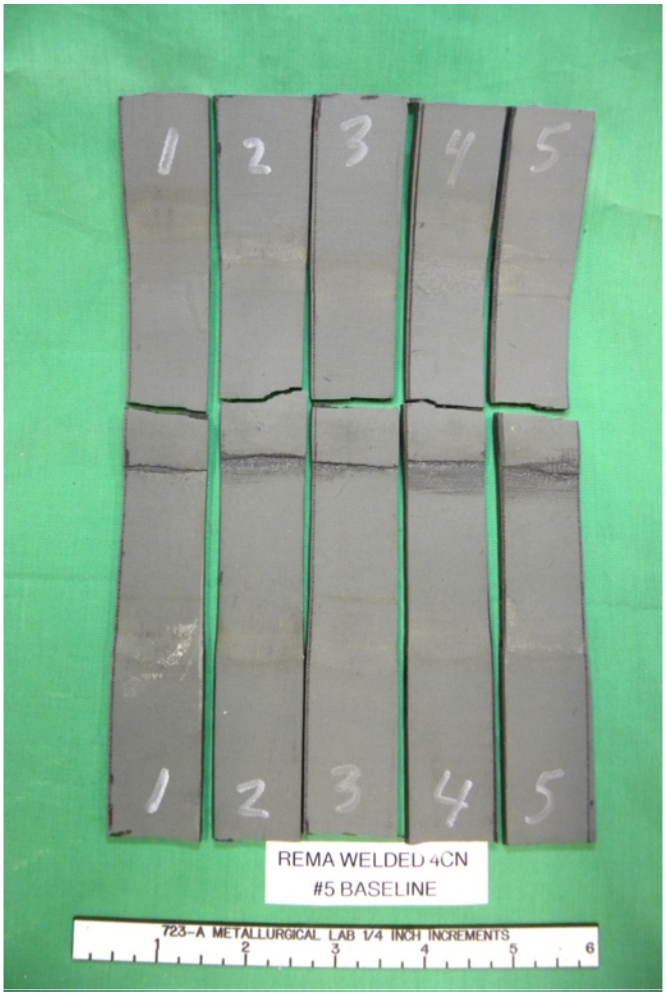

Figure 47. REMA 4CN lap-shear samples (baseline) – post-test (fractures in base material at joint edge)

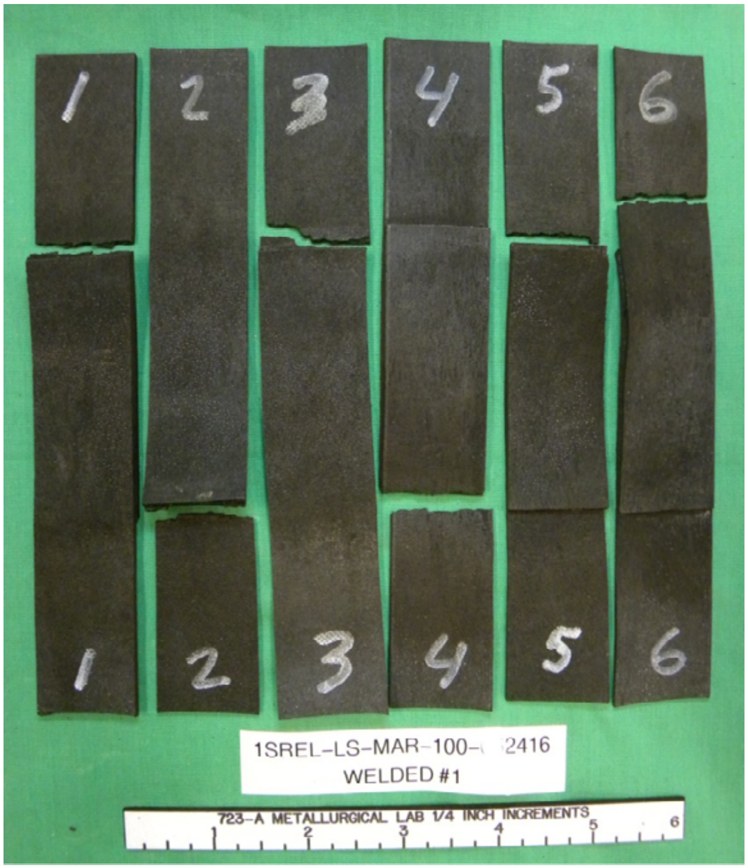

Figure 48. Marseal M-3500 lap-shear samples (100% S1 solution) – post-test (fractures in base material at joint edge)

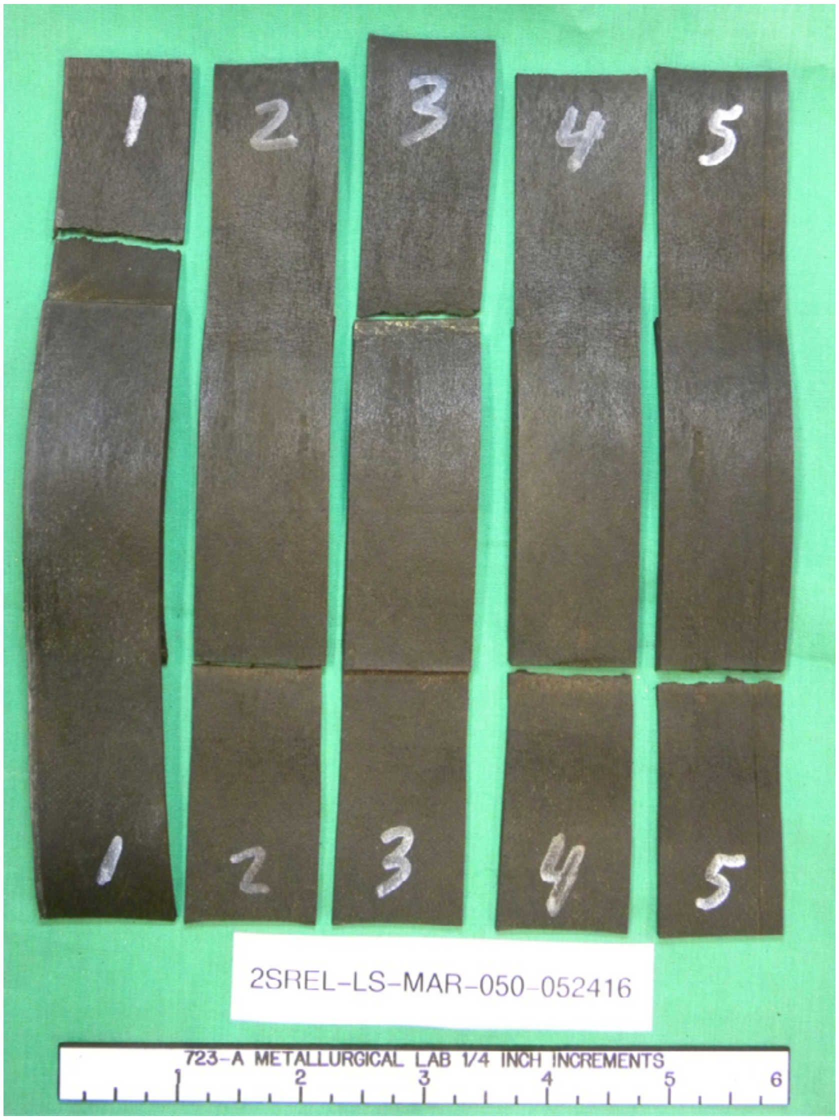

Figure 49. Marseal M-3500 lap-shear samples (50% S1 solution) – post-test (most fractures in base material at joint edge, #1 failed away from edge)

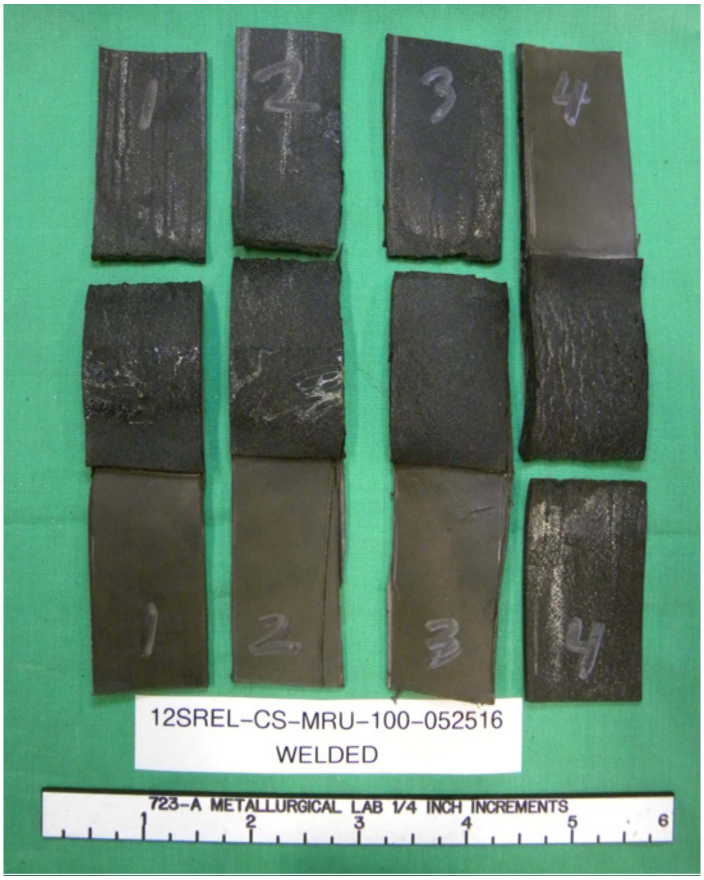

Figure 50. Marseal M-3500/Marseal 2000 lap-shear samples (100% S1 solution) – post-test (fractures in base material at joint edge)

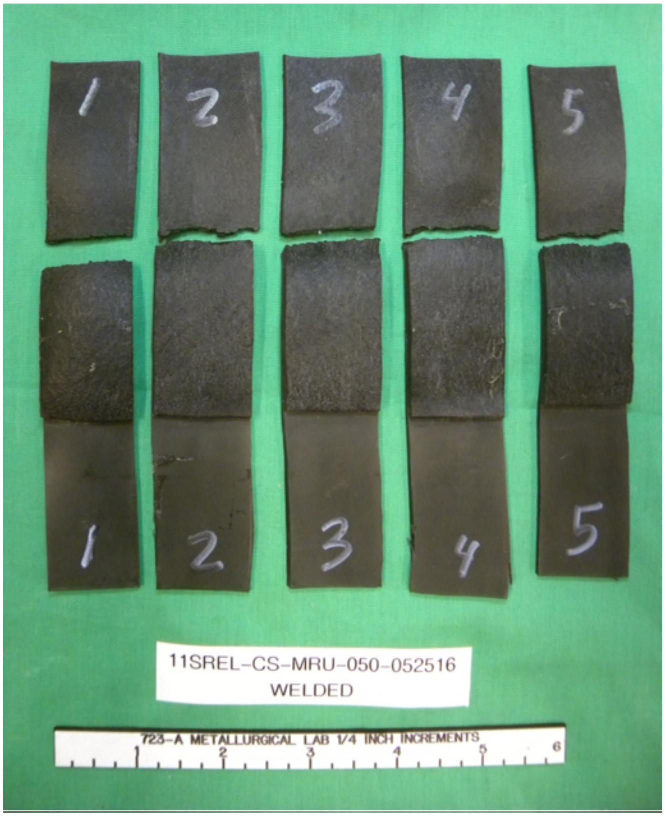

Figure 51. Marseal M-3500/Marseal 2000 lap-shear samples (50% S1 solution) – post-test (fractures in base material at joint edge)

Figure 52. REMA 4CN lap-shear samples (50% S1 solution) – post-test
(fractures in base material at joint edge or within joint)

Figure 53. REMA 4CN lap-shear samples (100% S1 solution) – post-test
(fractures in base material at joint edge or within joint)