

# ***FY21 Status Report: SNF Canister Coatings for Corrosion Prevention and Mitigation***

## **Spent Fuel and Waste Disposition**

***Prepared for  
US Department of Energy  
Spent Fuel and Waste Science and  
Technology***

***Andrew Knight, Brendan Nation,  
Charles Bryan, and Rebecca Schaller  
Sandia National Laboratories***

***September 3, 2021***

**M3SF-21SN010207062**

**Sandia National Laboratory Report No. SAND21-  
XXXXXXX**

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## **SUMMARY**

This report summarizes the current activities in FY21 related to the effort by Sandia National Laboratories to identify and test coating materials for the prevention, mitigation, and repair of spent nuclear fuel dry storage canisters against potential chloride-induced stress corrosion cracking. This work follows up on the details provided in Sandia National Laboratories FY20 report [1] on the same topic, which provided a detailed description of the specific coating properties desired for application and implementation on spent nuclear fuel canisters, as well as provided detail into several different coatings and their applicability to coat spent nuclear fuel canisters. In FY21, Sandia National Laboratories has engaged with private industry to create a Memorandum of Understanding and established a collaborative R&D program building off the analytical and laboratory capabilities at Sandia National Laboratories and the material design and synthesis capabilities of private industry. The resulting Memorandum of Understanding included four companies to date (Oxford Performance Materials, White Horse R&D, Luna Innovations, and Flora Coating) proposing six different coating technologies (polyetherketoneketone, modified polyimide/polyurea, modified phenolic resin, silane-based polyurethane hybrid with and without a Zn-rich primer, and a quasi-ceramic sol-gel polyurethane hybrid) to be tested, evaluated, and optimized for their potential use for this application. This report provides a detailed description of each of the coating systems proposed by the participating industry partners. It also provides a description of the planned experimental activities to be performed by Sandia National Laboratories including physical tests, electrochemical tests, and characterization methods. These analyses will be used to identify specific ways to further improve coating technologies toward their application and implementation on spent nuclear fuel canisters. In FY21, Sandia National Laboratories began baseline testing of the base metal material in accordance with activities of the Memorandum of Understanding. In FY22, Sandia National Laboratories will receive coated coupons from each of the participating industry partners and begin characterization, physical, and electrochemical testing following the test plan described herein.

This report fulfills the milestone "SNF Canister Coatings for Corrosion Prevention and Mitigation" (M3SF-21SN010207062) in the Canister Coatings for Prevention and Remediation work package (SF-21SN01020706).

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## **ACKNOWLEDGEMENTS**

The authors acknowledge the contributions to this report from Sandia personnel, Erin Karasz who performed the technical review, Sylvia Saltzstein and Emily Stein for additional review comments, and Lindsay McCabe, for help with editing and formatting. The authors would also like to thank Ken Sorenson and Jorge Monroe-Rammsy for their review along with Oxford Performance Materials, Whitehorse R&D, Luna Innovations, and Flora Coatings for their review comments and participation in this program.

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## **ACRONYMS**

CISCC	chloride-induced stress corrosion cracking
COTS	commercial off-the-shelf
CVCM	collected volatile condensable materials
DSC	dry storage canister
DOE	Department of Energy
DOD	Department of Defense
EDS	energy dispersive X-ray spectroscopy
EIS	electrical impedance spectroscopy
EPRI	Electric Power Research Institute
FC	Flora Coatings
FIB	focused ion beam
ISFSI	independent spent fuel storage installation
MOU	Memorandum of Understanding
NASA	National Aeronautics and Space Administration
NDE	non-destructive evaluation
NEUP	Nuclear Energy University Programs
OCP	open circuit potential
OPM	Oxford Performance Materials
PAEK	polyaryletherketone
PEEK	polyetheretherketone
PEKK	polyetherketoneketone
PNNL	Pacific Northwest National Laboratories
RH	relative humidity
SBIR	Small Business Innovation Research
SEM	scanning electron microscope
SNF	spent nuclear fuel
SNL	Sandia National Laboratories
TML	total mass loss
TRL	technology readiness level
WHRD	Whitehorse R&D

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## **SPENT FUEL AND WASTE SCIENCE AND TECHNOLOGY**

# **FY21 STATUS REPORT: SNF CANISTER COATINGS FOR CORROSION PREVENTION AND MITIGATION**

## **1. INTRODUCTION**

Exposure to spent nuclear fuel (SNF) can pose a hazard to the health and safety of the general population and of the environment. To prevent exposure, SNF is safely stored in dry cask storage systems at various independent spent fuel storage installations (ISFSIs) located across the United States (Figure 1) [2, 3]. In dry cask storage systems, the SNF assemblies are stored dry storage canisters (DSC). These DSCs are large ½ to 5/8-inch-thick welded stainless steel (SS) canisters (~6 ft in diameter and ~20 ft in length) and include the SS canister as well as fuel baskets, neutron absorber materials, and other structural components (Figure 2) [1]. The canisters are placed in concrete overpacks with vents for air flow that passively cool the SS canister. Together, the canisters and overpacks comprise the dry cask storage systems. The canisters can be stored in either vertical or horizontal configurations and can be located above ground or below ground. An example vertical, above-ground DSC is shown in Figure 2. Since the DSCs are passively cooled through air vents, dust, salt, and debris can accumulate on the canister surface over time. At sites where chloride (Cl<sup>-</sup>) salts exist (e.g., near-marine sites), it becomes possible for a corrosive brine to deliquesce on the surface of the canister as it cools over time. As storage times increase -- resulting in a decrease in surface temperature from radioactive decay -- and surface dust/salt loads increase, the potential for chloride-induced stress corrosion cracking (CISCC) increases. The consequence of penetration of the canister by CISCC would be increased risk of exposure to populations or the environment [4].

Ongoing work has focused on understanding the canister surface environment and its relationship to possible corrosion, pit-to-crack transition, and crack growth [1, 5-12]. While this work is important for prediction of when and where a crack may form, and how quickly a crack may grow, additional efforts have been initiated for the development of strategies to mitigate corrosion damage and extend the safe lifetime of SNF existing canisters. This study explores coating strategies to prevent, mitigate, and repair potential CISCC. In FY20, Sandia National Laboratories (SNL) produced a report summarizing a literature survey on possible common coating technologies for use on DSCs. In that report, SNL provided recommendations of promising potential coating strategies [1]. High interest technologies were chosen based on their chemical and physical properties, degradation resistance, and technology readiness level (TRL) for application on canisters. A preliminary test plan for coatings evaluation, to be carried out at SNL, was also included in the recommendations. In FY21, SNL engaged with industry experts in industrial coating technologies through a collaborative Memorandum of Understanding (MOU). The goal of this MOU was to develop an iterative R&D program to explore specific coating technologies for their use on SNF canisters. In selecting potential coating technologies, the primary focus was on corrosion prevention and possible use of coatings to stifle growth of existing SCC cracks, as presented in the framework described in the report from FY20. However, as both peening or burnishing techniques that rely on mitigation of weld residual stresses and metallic spray techniques such as thermal or cold sprays are currently under evaluation by several different Nuclear Energy University Program (NEUP) projects, and as part of a separate collaborative project between SNL and Pacific Northwest National Laboratories (PNNL), these technologies were not considered in the report or the industry MOU. Lastly, the work described in this report differs from the collaborative effort between SNL and Electric Power Research Institute (EPRI) that also plans to evaluate potential mitigation and repair techniques.

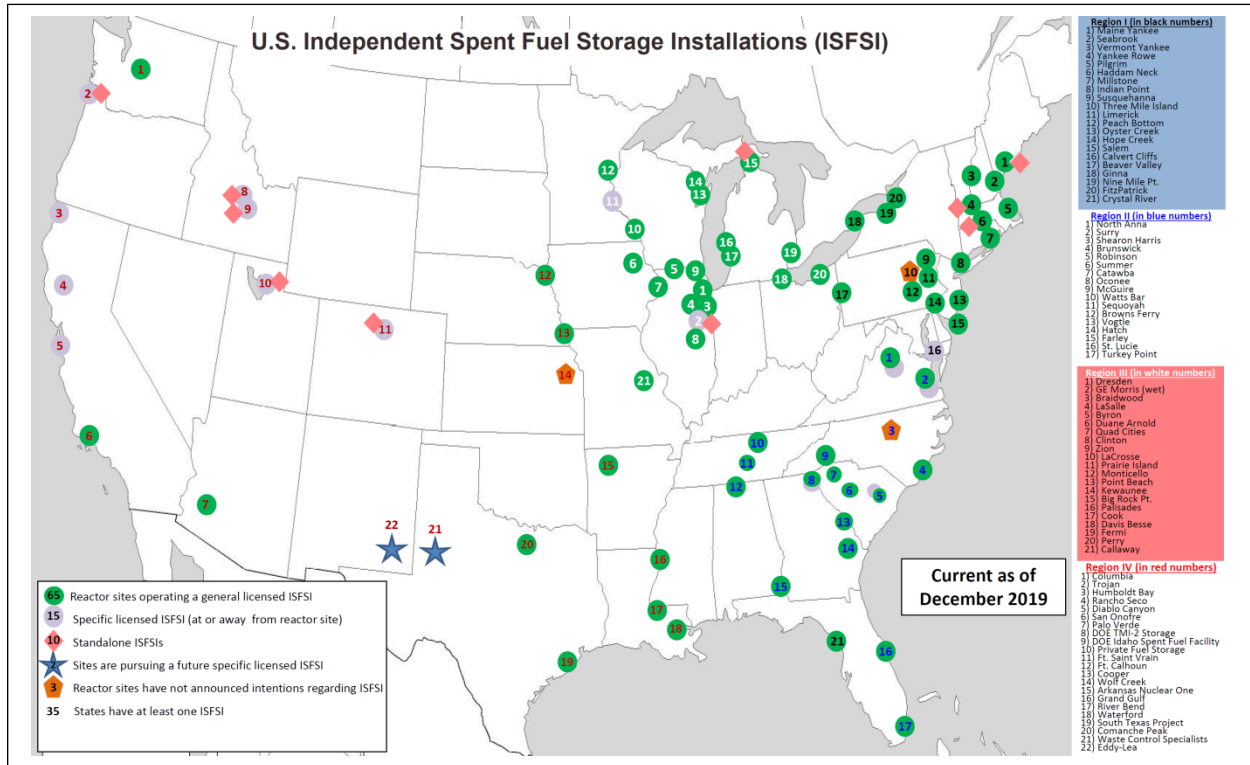


Figure 1. Map showing ISFSI sites in the United States [2].

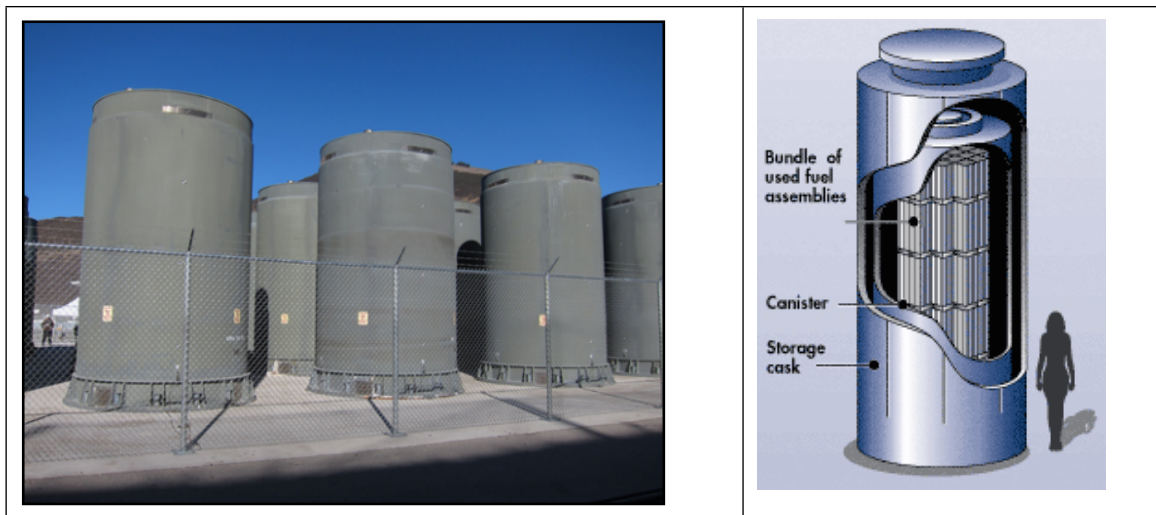


Figure 2. Left: Dry cask storage systems at the Diablo Canyon ISFSI (photo, C.R. Bryan); Right: diagram showing the structure of a dry cask storage system including SNF and the DSC within an overpack [13].

## 1.1 Specific Challenges and Requirements for Coatings

As a result of the FY20 SNL report [1], SNL has identified unique challenges that exist for selecting and applying corrosion resistant coatings to DSCs. Specifically, over the storage lifetime for SNF, the surface environment is in constant flux with respect to temperature, radiation, and potential corrosive

environment. In addition, due to the design of the dry cask storage systems, with the SNF and DSC inside an overpack or crypt, access and space limitations exist which create significant challenges to application of coating technologies.

### 1.1.1 Environment

For CISCC to occur, three criteria must be met. There must be 1) a susceptible material, 2) tensile stresses, and 3) a corrosive environment. Previous reports [1, 5-12, 14] have described in detail that in dry storage of SNF these three criteria will eventually be met, at least at some ISFSI sites. A susceptible material exists – most SNF canisters are 304 SS; weld residual tensile stresses are sufficient for CISC to occur; and a corrosive environment has the potential to form. During the storage lifetime of a SNF DSC, it is possible for a corrosive environment to develop; however, the severity of such an environment is location and time dependent. The greatest risk for CISC on SNF canisters occurs when a chloride-rich brine is present on the canister surface [1, 4-12, 14, 15]. This is most likely to occur from sea salt deposition – however other pathways for  $\text{Cl}^-$  salt deposition exist [7, 14]. Most ISFSIs are co-located with the nuclear power stations that generated the SNF; they are present throughout the entire United States (Figure 1), as result many ISFSI sites are located in near-marine settings. In near-marine environments, chloride-rich aerosols and salt particulates can be suspended by wind and may deposit onto the canister surface as air is drawn through the passively ventilated storage systems. Initially, the canister surface temperatures are too hot to allow a stable brine to form – therefore the surface remains dry, and the risk of corrosion and CISC are very low. However, as the canisters cool over time, the relative humidity (RH) increases to the point where salt deliquescence is possible. Once the deliquescence RH is reached and chloride is present, an aggressive chloride-rich brine can form on the canister surface. In the laboratory, sea-salt aerosols have been linked to corrosion—pitting and eventually CISC—of stainless steel [9-12].

The proposed strategy is to use coating materials to prevent, mitigate, and repair potential CISC, by minimizing or eliminating the interaction of a corrosive brine with the canister surface. To do so, an effective coating would eliminate the possibility of CISC by isolating the metal surface from the corrosive environment on the coated canister surface. Most coatings will either isolate the metal surface from exposure to aggressive chemistry by acting as a physical barrier limiting mass transport to the metal substrate or inhibit corrosion by use of corrosion inhibitors.

### 1.1.2 Implementation Strategies

In the FY20 SNL report, the three specific application scenarios where a coating could be implemented were defined as **ex situ prevention**, **ex situ repair**, and **in situ repair** [1]. The timing of coating application in the SNF canister life cycle is critical to define the necessary requirements and range of environments that the coating will be exposed to.

When coatings are applied as an **ex situ prevention** strategy, the coating material is applied to the canister surface during or just after manufacturing and prior to loading with SNF. In this situation, the coating serves as a corrosion barrier from the onset of the canister lifecycle. This situation allows for greater flexibility in application as the access limitations of a canister within an overpack do not apply, and hazards associated with a loaded canister (e.g., radiation), do not have to be considered. Therefore, applications methods that require baking, high temperature curing, or any other process that requires very precise control of the surrounding environment are possible (so long as the coating can be applied to a canister-sized surface). Application prior to loading SNF would allow for greatest access to the canister surface and greatest control of the environment; it also removes worker dose considerations. While this scenario is the most straightforward from an application perspective, the conditions that the coating will experience are the most extreme. The coatings must withstand the high temperatures (possibly 200 °C or higher) associated with the SNF loading, drying, and transportation in addition to the high levels of gamma radiation being emitted from the SNF during the early period of storage [1, 16]. The actual temperature range depends on a number of factors (e.g. initial heat load), which has been modeled previously [16]. The coating must also be robust enough to withstand potential mechanical impacts or

scrapes during canister filling and transfer operations. Note that, since the coating would be applied to the canister before the final closure weld is applied, that weld would not be protected; however, as the canister closure actually consists of two separate welded plates with a gap between, through-wall CISCC cannot occur, and the closure welds are not considered to need protection from CISCC.

A second implementation strategy is to apply coatings to the already existing canisters in order to prolong lifetimes through the protection and prevention of corrosion and potential CISCC. This was defined as **ex situ** or **in situ repair**. In the **ex situ repair** scenario, it is assumed that it is possible to remove the canister from the overpack and potentially transport it to a nearby controlled environment where a coating could be applied. One advantage for this type of implementation is that access to the canister surface is sufficient to allow for a larger variety of coating application techniques and more complete coating. A coating could be applied over the entire surface of the canister (including the welds), as a patch around areas of high risk (as determined by other studies), or as a patch in areas where corrosion has already initiated. Moreover, because the canister will be removed from the overpack, the canister can be thoroughly inspected to identify the extent of any existing corrosion, and properly cleaned in preparation for coating application. In contrast to the **ex situ prevention** strategy, some disadvantages of **ex situ repair** are the increased costs and risks associated with moving and coating an in-service SNF canister. It would be expected that the costs, both in terms of time and capital, would increase due to the restrictions imposed by working with a high radiation loaded canister. There would be the worker dose, the potential for accidents, and possible releases during the application process. There are likely also limitations as to what can be done to the canister to apply the coating (e.g. temperature control for curing of coating). A mechanically robust coating or a self-healing coating may also be necessary to mitigate possible damage during canister movement. This application scenario presents a significant opportunity to mitigate risk as there are thousands of canisters [17] already in use (some with greater risk of CISCC than others). A properly selected and applied coating could make a great impact on mitigating the risk of CISCC.

The last implementation scenario is the **in situ repair**. In this scenario, the canister must remain in the overpack due to restriction in removing it from site or safety restrictions associated with identification of a suspected crack or due to possible radiation exposure to workers. The coatings implemented in this scenario must be applied to the canister via remotely operated equipment inserted through the inlet or outlet vents without removing the canister from the overpack. An ideal coating for **in situ repair** could be applied and reapplied with minimal operational costs and can be used as a repair or prevention technique. Access limitations may have a large effect on implementation of any **in situ repair** coating technology. Further development of canister non-destructive evaluation (NDE) techniques are necessary to be able to deploy **in situ repair** techniques, as the current technologies are incapable of fully assessing corrosion damage or identify cracks. Further, coatings applied in this scenario would likely not be applied over the entire surface of the canister due to access limitations to certain regions in the overpack (i.e. the canister ends and underneath the support rails). Due to this limitation, either methods for coating access limited areas are required or coatings in this scenario should probably only be considered as “patches” to treat localized, accessible regions.

Further discussion regarding these scenarios is provided in the FY20 report, including a table that highlights specific coating requirements for each case. In addition, the FY20 report provides a summary of additional details regarding different characteristics that may be needed or desirable for a coating to be effective for use on SNF DSCs.

## 1.2 Conclusions from FY20 Report

In the FY20 report, SNL summarizes several classes of coatings that provide potential solutions to one or more of the prevent, mitigation, and repair scenarios for preventing, sealing, or arresting potential CISCC. Specifically, polymeric, ceramic, and conversion coating types were considered, in addition to a discussion on the current work on kinetic spray coatings (PNNL/SNL), such as cold spray [1, 11]. A summary of the conclusions of the FY20 report is shown in Table 1, where the coating technologies are



color-coded to indicate possible usefulness. In this case, red cells indicate that the specific coating property is not amenable for use in the coatings current state, orange cells indicate that coating properties suggest that the coating may be possible but requires additional study, and green cells indicate that the coatings' properties could be used with little or no modifications or development. From these results, five coating technologies were identified as promising candidates for further study, shown in Table 1 with an asterisk.

### 1.2.1 Summary of Initial Coating Recommendations

The recommendations from FY20 are summarized below.

- **Polymer Coatings:** Polymer coatings that demonstrated the most promise for use on SNF canisters were epoxies, polyethylene coatings, and rubbers (Table 1). These conclusions were made based on the properties of these coatings, their current TRL level, their ubiquitous use throughout the industry, and their ease of application. This makes them potentially desirable for an **in situ repair** applications as all three types can be brushed on – a technique that could be easily developed for robotic application. A potential challenge for these techniques is that they typically require surface preparation procedures that may be limited or impossible to perform on **in situ** DSC. Research is necessary to evaluate the effects of existing rust/contamination and metal surface roughness on coating adhesion. Each of these polymer coatings could be used for **ex situ** applications; however, research must identify their thermal and radiation stability under realistic exposure conditions. Generally, rubber coatings could be the best candidate for **ex situ** applications because they have relatively high thermal stability (250 to 600 °C), but selection of a specific type of rubber to match the canister thermal conditions would be necessary. Lastly, studies of mechanical robustness and possible reapplication over a scratched or damaged polymer coating are required to determine robustness and feasibility of use canister transfer or transport scenarios.
- **Ceramic Coatings:** Ceramic coatings using sol-gel synthesis are believed to have potential use in both **in situ** and **ex situ** scenarios due to their great chemical, thermal, and radiological stabilities. A benefit of sol-gel coatings is their versatility. Many sol-gel properties depend upon the metal-oxide used in the sol and on alterations to the coating structure made by the addition of additives (e.g. polymers). Current technology limits sol-gel implementation to **ex-situ** strategies; however recent work has demonstrated sol-gel application via spray or brushing techniques. The availability of these techniques improves the feasibility of ceramic coatings for use as a base layer for polymer coatings.
- **Conversion Coatings:** Phosphate conversion coatings have potential to be used on SNF canisters due to their mechanical, chemical, and thermal stability. They have been used for corrosion protection on steel and stainless steels. They are of particular interest for use as an **ex situ** coating – either by themselves, or as an undercoat for polymer coatings to improve the adhesion and corrosion resistance properties of the polymer. It is unlikely or impossible to apply phosphate conversion coatings to an SNF canister **in situ** because application is a complex multistep process. It was recommended that phosphate conversion coatings be evaluated for use as a corrosion prevention treatment and applied prior to loading SNF.

The analysis in FY20 laid the framework for how to address some of the specific criteria required for a coating to be used to either prevent, mitigate, or repair potential canister CISC. While many coating types were evaluated, this was not an exhaustive list of all available coatings. The coatings industry is a large field that primarily exists in the private sector, and advancements are made using proprietary innovations to specific coatings in order to improve their viability for specific applications. For this reason, using the knowledge gained during the FY20 analysis, SNL engaged with coating manufacturing companies in the private sector to advance an MOU R&D program to address possible coating solutions

for **ex situ prevention, ex situ repair, or in situ repair** scenarios. This work is complementary to the ongoing collaborative work between SNL and PNNL to advance cold spray technologies [10, 11] (FY21 details are addressed in M2SF-21SN010207056) which will be compared to the identified solutions in this report. This report fulfills the milestone "SNF Canister Coatings for Corrosion Prevention and Mitigation" (M3SF-21SN010207062) in the Canister Coatings for Prevention and Remediation work package (SF-21SN01020706).

**Table 1. Summary of FY20 conclusion addressing important coating properties and potential uses for SNF dry storage canisters [1].**

Coating		Attribute	Implementation		
Class	Coating Name	Properties/Degradation	In situ repair	Ex situ repair	Ex situ prevention
Polymer	Air Dry Epoxy *	Susceptible to radiolytic degradation; not stable above 130°C	Minimal surface preparation; Requires T< 130° C	Minimal surface preparation; Requires T< 130° C	Susceptible to radiolytic degradation; Requires T< 130° C
	Polyvinylidene Fluoride	Thermally stable but thermal expansion mismatch; heavily prone to scratching	Prone to scratching, required; Must be cured at 260 °C – 360°C	Prone to scratching, required; Must be cured at 260 °C – 360°C	Mismatch in thermal expansion
	Graphene Enhanced Epoxy	Improved properties over air dry epoxies	Requires spin coating	Requires spin coating	Requires spin coating;
	Polyurethane	Thermally, chemically, and radiolytically stable; Prone to blistering and requires frequent reapplication	Thermal expansion issues increase risk of blistering; requires frequent reapplication	Thermal expansion issues increase risk of blistering; requires frequent reapplication	Thermal expansion increase risk of blistering; requires frequent reapplication
	Polysiloxane	Chemically and mechanically stable; Sensitive thermal and radiolytic stability; Frequent reapplication	Minimal surface and application requirements, but require frequent reapplication	Minimal surface and application requirements, but require frequent reapplication	Poor thermal and radiolytic stability
	Polyethylene *	Chemically and mechanically stable; radiolytically sensitive; unknown thermally; multiple layers application can increase time to degradation	Can be easily applied as short term patch due potential radiolytically degradation	Can be easily applied as short term patch due potential radiolytically degradation	Poor radiolytic stability
	Alkyd Paint	Poor radiolytic and mechanical stability; primers are needed for adhesion	Can be applied easily, but requires primer	Poor mechanical robustness	Poor radiolytic stability; poor mechanical robustness
	Polyvinyl Chloride	Thermally degrades above 60°C; poor radiolytic stability	Low temperature range makes application impossible (<60°C)	Low temperature range makes application impossible (<60°C)	Low temperature range makes application impossible (<60°C)
	Polyester	Robust chemically and mechanically, sensitive to thermal and radiolytic breakdown; deposition method is not possible on a canister	Electrostatically spun on at 180°C	Electrostatically spun on at 180°C	Electrostatically spun on at 180°C; Prone to thermal and radiolytic degradation
	Rubber *	Robust but susceptible to permeation but can be improved with multiple layers; stable to high temperatures	Can be painted or sprayed on	Can be painted or sprayed on	Can be painted or sprayed on
Ceramic	Sol-gel *	Chemically, thermally, radiolytically and mechanically stable; adhesion and application depends on additives and surface finish, prone to brittle failure	Can be applied by spray or brush methods	Prone to scratching and brittle failure, but can be improved with additives	Prone to scratching and brittle failure, but can be improved with additives
	Ion Beam Assisted Deposition	Chemically, thermally, radiolytically and mechanically; great adhesion; prone to brittle failure	Requires special equipment	Requires special equipment	Requires special equipment
	Chemical Vapor Deposition	Chemically, thermally, radiolytically and mechanically; great adhesion; prone to brittle failure	Requires special equipment	Requires special equipment	Requires special equipment
Conversion	Chromate Conversion	Chemically, thermally, radiolytically and mechanically stable; Requires a surface plated with Al or Zn; No evidence of success bare SS surface	Hazardous; No evidence of success on SS	Hazardous; No evidence of success on SS	Hazardous; No evidence of success on SS
	Phosphate Conversion*	Chemically, thermally, radiolytically and mechanically stable; great adhesion; Complex application and reapplication process	Complex application and reapplication process	Complex application and reapplication process	Effective coating if applied during prior to SNF fuel loading
Spray	Cold spray	Robust and great adhesion; surface modification effects on corrosion must be demonstrated	Can be applied locally with robotic crawler	Can easily be applied locally	Can be easily applied

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## **2. FY21 STATUS AND INDUSTRY PARTICIPATION**

In FY21, SNL engaged with the industry partners to develop a collaborative research endeavor that capitalizes on the scientific and testing capabilities at SNL and decades of industry expertise in the development of corrosion resistant coatings. The demands on coatings to prevent, mitigate, or repair corrosion and potential CISCC are not new, and apply to many different applications (e.g., aerospace and naval equipment). Pursuing a collaboration between SNL and industrial partners allows for SNL to leverage commercial proficiency to develop coatings geared toward applications involving DSCs. While corrosion resistant coatings are not new, to date there have been no coatings applied to SNF DSCs. And, as described in detail in Section 1.1 and the FY20 report, the use of corrosion resistant coatings to prevent, mitigate, or repair potential CISCC on SNF canisters has specific limitations that have no clear analog to other commercial examples. For this reason, SNL has solicited the interest of several coating manufacturers to tailor their coating technologies to address the specific requirements for use on an SNF canister, through the implementation scenarios described above.

To create a collaborative research structure between SNL and the industry partners, SNL issued a solicitation describing the desired coating needs for application on SNF DSCs. Coating companies were then able to respond to the call and submit coating technologies as well as a justification for how the proposed coating can be used as a mitigation and repair strategy against CISCC. The solicitation was made public as a federal business opportunity (FBO) on the SAM.gov website. SNL then selected coating technologies that either matched the desired coating types from the FY20 report (e.g. sol-gel coatings), or for which the coating company was able to provide a justification showing that it met the specific requirements. Upon selection of a particular coating technology, SNL developed an MOU with the coating manufacturer to engage in a collaborative research effort. The MOU is a legal partnership structure which allows maximum flexibility between SNL and industry partners while also allowing direct collaboration between the involved entities. As discussed previously, the FY20 report was not a comprehensive assessment of all coating possibilities, but rather a development of scope and the desirable characteristics for a coating to be applied in this context. Therefore, the current selected coating types reflect in part those described in FY20 with additional types (as suggested by industry) that were not previously discussed. Further, certain types of coatings (e.g. rubber, phosphate conversion coatings) were not proposed by any commercial coating manufacturer to date, and therefore are not included in the current list of proposed coatings. However, as the design of this SNL/industry MOU is open-ended, this allows for SNL to add or remove coating types throughout the project based on performance results, coating characteristics discovered during the collaboration, or inclusion of new, desirable coatings outside of the scope of the current MOU.

### **2.1 Details of the Memorandum of Understanding**

SNL has established collaboration through the MOU with Oxford Performance Materials (OPM) to evaluate a modified polyetherketoneketone (PEKK), OXPEKK®-SC; Whitehorse R&D (WHRD) to evaluate a modified polyimide/polyurea (CrakStop®), modified polyimide/polyurea/phenolic resin (GammaBlock®), and a modified phenolic resin (GammaBlock Plus®); Luna Innovations (LUNA) to evaluate Gentoo™, a hybrid organic-inorganic coating matrix based on a silane modified polyurethane to be used, with or without a zinc-rich primer; and Flora Coatings (FC) to evaluate CLADCO™, a hybrid quasi-ceramic silica-based sol-gel ceramic coating modified with a single component polyurethane polymer linker. The goal is to determine the ability of each coating to meet the requirements for implementation on SNF canisters for **in-situ** and/or **ex-situ** prevention and repair strategies. Specific details of the coating technologies are provided in Section 0.

The objective of this MOU is to determine if coating technologies proposed by OPM, WHRD, LUNA, and FC are viable corrosion resistant coatings. A successful candidate coating will provide adequate protection against moisture permeation, adhere well to the substrate surface, be durable enough to resist

abrasion damage, and in doing so, provide protection against corrosion processes. Additionally, these material properties will not degrade significantly with temperature and radiation over their lifetime in relevant exposure conditions. The advantage of establishing an MOU is that SNL has become a leader in the development of a wholistic understanding of the risks CISC of SNF canisters through an in-depth understanding of the surface environment and its impact on localized corrosion and potential CISC. In doing so, SNL has demonstrated a need for the development of corrosion-resistant coatings for mitigation and repair of SNF dry storage canisters. OPM, WHRD, LUNA, and FC have developed expertise and materials to address similar problems. Collectively SNL with OPM, WHRD, LUNA, and FC will apply coatings to stainless steel coupons for SNL to experimentally evaluate.

The MOU describes the planned work in three phases. The phases evaluate coating technologies with increasingly aggressive exposure conditions and surface challenges (e.g. a corroded surface with corrosion products). A brief description of each phase is described below.

- **Phase 1:** The goal of Phase 1 is to provide a baseline understanding of the coating physical, mechanical, and electrochemical properties under ideal conditions. These tests will be the baseline for continued collaboration between SNL and OPM, WHRD, LUNA, and FC to iterate, tune, and develop coating technologies for CISC prevention, mitigation, and repair in SNF canisters. Pristine SS coupons will be coated by OPM, WHRD, LUNA, and FC using several application methods. The quality of the coating will be assessed by physical characterization (e.g. adhesion pull-off testing, scratch tests) and chemical/electrochemical testing (e.g. water permeability), conforming to publicly available ASTM standards, when applicable. Corrosion exposures under canister-relevant conditions will be performed, and physical and chemical tests will be performed again to determine the impact of a corrosive environment on the coating performance. The purpose of Phase 1 is to identify the best candidate coating materials and their manufacturing and application processes. Success is defined as having good overall performance in initial tests without any detrimental chemical, physical or mechanical properties. Coatings with the best performance will be carried forward to Phase 2.
- **Phase 2:** The goal of Phase 2 is to develop an understanding of the radiation and thermal stability of the coatings—how exposure to gamma and thermal radiation will impact the performance of the coatings. This phase focuses on coating application under **ex situ prevention** and **ex situ repair** scenarios where radiation, thermal, and mechanical environments are most extreme. More rigorous performance testing will be performed. Again, pristine coupons will be provided to industry partners, and they will apply proposed coatings to the surfaces. These coatings may vary somewhat from those applied in Phase 1, having been further optimized for this application based on results from Phase 1. These coupons will be exposed to different environmental conditions to evaluate their thermal, radiation, chemical, and mechanical resistance (conditions similar in magnitude and severity to expected canister-relevant conditions). In addition, corrosion experiments will be conducted to evaluate corrosion performance relative to control, uncoated SS samples and to coated samples that have not been exposed to heat or radiation.
- **Phase 3:** The goal of Phase 3 is to apply the previously successful coatings, from Phases 1 & 2, under non-ideal, canister-relevant surface conditions. Because technologies are desired to repair/mitigate corrosion on fielded SNF canisters, the surface of the SNF canister may not be pristine at the time of coating application. Coatings applied during Phase 3 target the **in situ repair** implementation scenario, where the canister surface may have significant dust or corrosion product and cleaning procedures may be limited due access limitations within overpacks. Coupons will be exposed to an environment that results in a corroded, contaminated (with dust or salt) and/or damaged (surface roughness alterations, cracks, welds, thermal sensitization, or some combination) surface. The surfaces will be characterized and sent to OPM, WHRD, LUNA, and FC to coat. Under these conditions, different surface preparation methods (such as cleaning, abrasion, etc) will be tested and documented, and subsequently evaluated in a

similar manner to the tests described in Phase 1 & 2. In addition, corrosion exposure studies under canister relevant conditions will also be performed.

In FY21, SNL has completed the MOU with the industry partners described herein and has started Phase 1 by performing initial baseline testing and providing the coupons to the industry partners.

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### 3. COATING TYPES

Through the MOU process SNL has identified several coating types and industry partners that have the potential to be used on SNF canisters. These coatings and their manufacturers are listed in Table 2 along with their coating class, possible application method, and based upon initial research, what implementation scenario the coating may be applied to the canister.

The FY20 report identified that air-dried epoxy, rubber, polyethylene, sol-gel, and phosphate conversion coatings were most applicable of the coating types evaluated. The current selection of proposed coatings only features one coating type from the FY20 report (sol-gel coatings) because there were no interested industry partners to date that proposed using air-dried epoxies, polyethylene, or phosphate conversion coatings. However, other polymer coatings were suggested such as a PEKK thermoplastic, a modified polyimide/polyurea resin, and a phenolic resin; all of which have many positive attributes which make them potentially viable for this application.

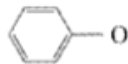
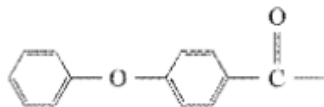
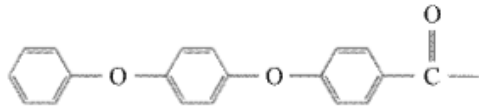
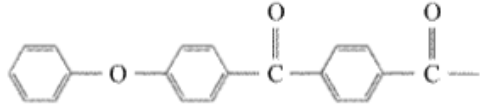
**Table 2. Details of the proposed coatings to be tested**

Coating	Vendor	Trade Name	TRL <sup>a</sup>	Coating Class	In Situ Repair	Ex Situ Repair	Ex Situ Prevention	Application Method
<b>Polyetherketone ketone (PEKK)</b>	OPM	OXPEKK®-SC; OXPEKK®	8/9	Polymer/ Thermoplastic	X	X	X	Solution or Powder
<b>Polyimide/Polyurea Resin</b>	WHRD	CrakStop®	9	Polymer	X	X	X	Paint, Roll, Spray, Pour
<b>Polyimide/Polyurea /Phenolic Resin</b>	WHRD	GammaBlock®	9	Polymer	X	X	X	Paint, Roll, Spray, Pour
<b>Phenolic Resin</b>	WHRD	GammaBlock Plus ®	9	Polymer	X	X	X	Paint, Roll, Spray, Pour
<b>Organic-inorganic hybrid matrix based on a silane-modified polyurethane with a Zn-rich Primer</b>	LUNA	Gentoo™ + COTS Zn-rich Primer	7/8	Sol-gel /polymer hybrid/ galvanic primer	X	X	X	Paint, Spray, roll
<b>Organic-inorganic hybrid matrix based on a silane-modified polyurethane</b>	LUNA	Gentoo™	7/8	Sol-gel/polymer hybrid	X	X	X	Paint, Spray, roll
<b>Hydrophobic inorganic polymer/quasi-ceramic</b>	FC	CLADCO™	8	Sol-gel/ polymer hybrid	X	X		Paint, roll, brush

<sup>a</sup> TRL levels are general and were provided by the collaborating industry partners. These TRL levels do not reflect their readiness toward coating SNF canister as this has not yet been demonstrated.

### 3.1 OX-PEKK®

PEKK belongs to the polyaryletherketone (PAEK) family of polymers [18, 19]. These polymers are well recognized as anti-corrosion materials [18, 19]. The general monomer chain for a PEKK is shown in Figure 3, which can be rendered reversibly water-soluble via functionalization of the aryl-groups (OXPEKK®-SC). PEKK is a semi-crystalline polymer material with robust chemical, thermal, radiation, and mechanical properties. PEKK-based materials have been used in environments ranging from -150 °C to 150 °C and have been used in a wide variety of applications, such as biomedical, automotive, and energy production. OPM has shown that OXPEKK®-SC (a modified PEKK coating developed by OPM) has great chemical resistance, which has been demonstrated by OPM in different types of aggressive exposure environments followed by electrochemical measurements to estimate the amount of degradation caused by the environmental exposures. OXPEKK® maybe also applied in native form, using common powder coating processes. In addition, PEKK has notable adhesion properties, even when compared with other PAEK materials. This has been attributed to the fact that PEKK exists at the interface between an amorphous and semi-crystalline material where it enables load transfer and increase continuity between the PEKK and the substrate [20, 21]. The radiation resistance of PAEK class polymers is high. This stability is achieved through the resonance stabilization in the aryl groups of radicals that may form because of exposure to ionizing radiation. This property has been demonstrated with polyetheretherketone (PEEK), known for its use in the nuclear industry; it has shown a radiation tolerance of to 10 mega Gray (MGy) [22, 23]. Due to the increased percent ketone functionalization (Figure 3) of PEKK over PEEK, it is expected that the radiation tolerance of PEKK is even greater.

Structure	Name	Ketone (%)
	PPO	0
	PEK	50
	PEEK	33
	PEKK	67

**Figure 3. Common monomer structure of several PAEK based polymers, including PEKK and PEEK [20, 24].**

OPM proposed two viable PEKK coating technologies for creating a corrosion-resistant coating on SNF canisters. These PEKK variants are a powder coating (Figure 4) (either of electrostatic and fluidized bed), thermal (fuel/flame) spray (if available at time of test) and solution dip (OXPEKK®-SC). These have undergone extensive development efforts over the last 20 years by OPM. OPM has previous experience working with the DOE and the DOD (Army Research Lab) to create workable solutions to corrosion-related issues. The commercial supply chains already exist to supply the base polymer(s) for the powder

coating and thermal spray variants. Vendors have been identified and discussions have been initiated to develop a commercial supply chain to provide solution materials. PEKK has the potential to be an effective polymer material for this coating application because it is highly corrosion and radiation resistant compared to conventional polymer coating technologies.

### OXPEKK® Powder Coating: Samples



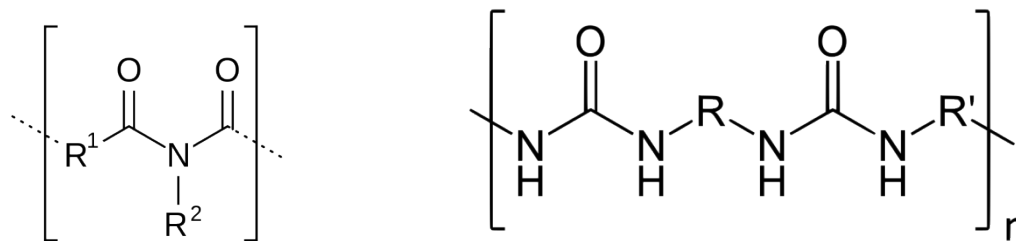
Figure 4. Examples of materials coated with OX-PEKK® provided by OPM.

Application options for PEKK can be varied and OPM plans to explore two methods that may be suitable for use on SNF canisters. Both powder and solution spray variants of PEKK have potential to be applicable for both **ex situ prevention** or **ex situ repair** type applications – and possibly **in situ repair**. However, the high temperatures of canister surfaces during and immediately after loading may limit its applicability as an **ex situ prevention** coating. SNL will work with OPM to direct the specific implementation requirements of each scenario to determine how and where each of the methods could be implemented. While these application methods (electrostatic powder coat and solution dip) have seen recent success, OPM has identified additional viable alternatives for development if the need should arise to pivot or further tailor PEKK coatings for application on SNF DSCs.

## 3.2 CrakStop® and GammaBlock®

A modified polyimide-polyurea resin consists of both polyimide and polyurea monomer groups with additional functionalization to allow for property tunability to target specific coating properties (Figure 5). As individual coating classes, both polyimides and polyurea are widely used industrially. Polyimides are lightweight materials that have great flexibility, thermal stability, and chemical stability and are used for many industrial applications, such as electronics and aerospace [25, 26]. Their use is widespread because polyimide-based coatings have superior properties to many other polymer coatings. Some of these specific properties include a large service temperature range (-300 °C to 300 °C), enhanced electrical insulation (dielectric constant = 3.4 – 3.5), and robust tensile strength after exposure to high thermal and radiation exposures [26-28]. Polyurea coatings, which are chemically similar to polyimide-based coatings, are well established and widely used in many industrial applications. Some of the positive characteristics of polyurea coatings are attributed to having a highly developed H-bonding network that both enhances the coating barrier properties by reducing the porosity and increasing the mechanical strength [29].

Limitations exist for polyimide and polyurea coatings; specifically, polyimide coatings can adsorb moisture, leading to poor adhesion and interfacial failure on some materials [30]. However, recent work has demonstrated that a novel class of copolymers, consisting of polyimide and polyurea blocks, improve many of these properties are capable of being effective corrosion resistant polymers for many applications; specifically, they been shown to be effective on 2024-T3 aluminum in saturated NaCl for over 30 days [30]. One reason for the effective synergy between polyimide and polyurea copolymers is that polyimides provide an H-bonding acceptor to the polyurea H-bonding donor. This results in precisely-controlled intra- and inter-hydrogen bonding and can result in induced self-assembly [30-32]. The impact of the enhanced H-bonding interactions is a reduction in surface energy, yielding a more compact morphology that improves the coatings ability to be impermeable to ionic corrosive species [30].

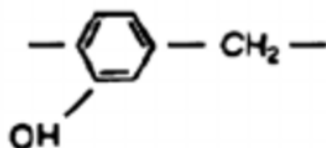


**Figure 5. General monomer structure of polyimide (left) and polyurea (right). The proposed solutions by WHRD (CrakStop® and GammaBlock®) involves a modified resin containing these polymers.**

WHRD proposes to use a highly modified polyimide/polyurea-based resin coating system. The modifications of the co-polymers allow for the coating to be tuned to provide a match for 304L SS (or the oxide coatings on them) when the surface temperatures are lower than ~120 °C (~250 °F) –specifically targeting **ex situ or in situ repair** implementation scenarios. The proposed resins will adhere and elongate to handle temperature change induced strains caused by coefficient of thermal expansion (CTE) mismatches between the coatings and the steel substrate. According to WHRD, these coating systems are designed create a hermetic seal to stop diffusion of oxygen, chlorine, sodium chloride, magnesium chloride and other chlorides; are expected to handle gamma radiation without degradation; and remain chemically stable while maintaining cohesive strength. Additional modifications targeting specific improvements can be achieved using additives such as fibers, solids, and other chemical modifiers. WHRD proposed that the modified polyimide/polyurea coating system may arrest known CISC crack by eliminating oxygen and chloride access to the crack. WHRD will coat samples with CrakStop® and GammaBlock®, where GammaBlock® will incorporate functionality from CrakStop® and GammaBlock Plus® (described in detail in Section **Error! Reference source not found.**), which would increase the radiation resistance of the coating through resonance stabilization of the incorporated phenol rings.

### 3.3 GammaBlock Plus®

Phenolic resin materials consist of a phenol monomer (Figure 6) [33-35]. Phenolic resins are very common and have been used for industrial applications for many decades [33, 35]. Phenolic resins have been used as a corrosion resistance coating and have demonstrated excellent thermal resistance (>300 °C) and chemical resistance to salts, non-oxidizing inorganic acids, and other solvents, however, unmodified phenolic resins are brittle and do not perform well under mechanical stress [35]. Initial efforts to improve the mechanical properties, including the creation of alkyl-phenolic resins, resulted in a more flexible material but also less chemical resistance [35]. More recently, the combination of phenolic resins with other modifiers (e.g. carbon nano-tubes), has been shown to improve both mechanical and chemical resistance [34].



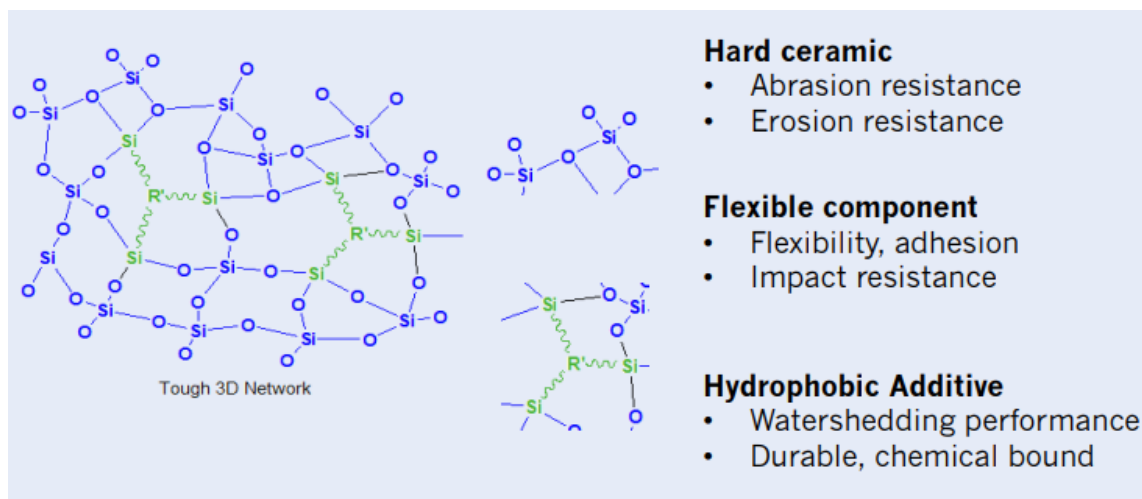
**Figure 6. General monomer structure of a phenolic resin used in GammaBlock *Plus*® [34].**

WHRD is proposing to use a high-temperature phenolic resin-based coating, GammaBlock *Plus*®, to reduce the risk of CISC on in service SNF canisters for **ex situ repair** and **prevention** applications. The specific coatings (modified phenolic resins) proposed by WHRD have been extensively used in “down hole” environments for fracking and oil exploration and have demonstrated efficacy on thousands of parts and components for tubing, pipelines and valves. WHRD in collaboration with SNL will work to determine which modifications will be required to create a phenolic resin based GammaBlock *Plus*® with the desired properties for the prevention, mitigation, or repair of potential CISC on SNF canisters. The specific properties that can be tuned are glass transition temperature ( $T_g$ ), adhesion, density, and mechanical toughness. WHRD has worked in the past with DOE and DOD on other projects to develop corrosion resistant coatings tailored for specific applications.

### 3.4 Gentoo™

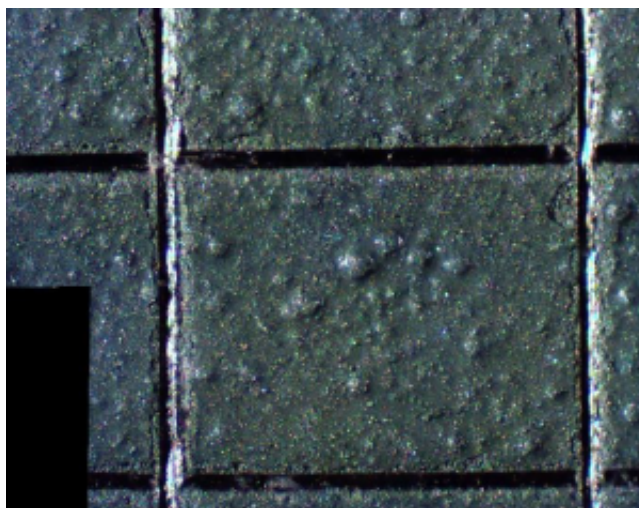
The Gentoo™ coating proposed by LUNA is an organic/inorganic hybrid coating consisting of a hard-ceramic component coupled with a flexible linker to produce high toughness for enhanced abrasion resistance and durability. Specifically, Gentoo™ is comprised of a transparent, organic-inorganic hybrid matrix based on a silane-modified polyurethane. The coating chemistry incorporated into Gentoo™ creates a material platform that results in a tough coating with high abrasion resistance, durability, and corrosion protection. The hydrophobic, polyurethane additives are incorporated for increasing water shedding and self-cleaning properties and allow for the tunability of properties; specifically, hardness, flexibility, and modulus to meet specific needs of an application (e.g. adhesion, abrasion, and weatherability). The generic structure of Gentoo™ is provided in Figure 7, where the  $\text{SiO}_2$  hard ceramic groups are linked together with a flexible, hydrophobic polymer creating a tough 3-D coating network. This type of sol-gel/polymer hybrid coating is specifically mentioned in the FY20 SNL report on coatings as a potential solution for use on SNF canister in a variety of implementation scenarios [1] – suggesting that many of sol-gel coating properties (e.g. permeability, mechanical robustness) can be improved by adding a polymeric linker. However, one potential pitfall of incorporating polymeric linkers into sol-gel coatings is that, depending on the identity of the polymer, it can reduce the radiation stability of the coating. In addition to performance testing in *Phase 1*, Gentoo™ will be rigorously evaluated in *Phase 2* for its thermal and radiation stability to ensure that the addition of a polymeric linker does not reduce the usefulness of the sol-gel coating for this application.





**Figure 7. General chemical structure, provided by LUNA, of the modified sol-gel coating, Gentoo™, proposed by LUNA.**

Another component that can be incorporated into the Gentoo™ coating is to apply it with a commercial off-the-shelf (COTS) Zn-rich primer. The addition of Gentoo™ to a COTS Zn-rich primer has been shown to improve the adhesion properties of the primer and therefore creates a more robust coating system. In addition, a Zn-rich primer would provide galvanic protection. This protection is created by the formation of a galvanic cell, in which Zn is a more active metal than SS. When damaged or exposed to a corrosive environment, Zn will react before the SS and precipitate to form a Zn-oxide protective coating on the surface of the SS to offer protection. The option to combine Gentoo™ with a Zn-rich primer allows for another layer of protection, that would aid in the prevention, mitigation, or repair of CISCC. This strategy would passivate the metal surface with galvanic coating and also provide a barrier through the addition of a protective, hydrophobic topcoat. With the use of a Zn-rich primer, specific evaluation of the potential for hydrogen embrittlement to occur must be performed, as it has been demonstrated that Zn-rich primers, in some cases, may cause overly cathodic polarizations and generate hydrogen [36]. Therefore, in this case, SNL will assess whether the use of a Zn-rich primer adds additional benefit to the viability of Gentoo™ for use on SNF DSCs or if its use has the potential to cause hydrogen embrittlement.



**Figure 8.(Left) Example of a material coated with a COTS Zn-rich primer with Gentoo™.**

LUNA proposed the use of Gentoo™ with and without the COTS Zn-rich primer as a solution for the prevention, mitigation, or repair of SNF canisters and has the potential to implement this during **in situ repair** and **ex situ repair** scenarios. Depending on the properties of the organic component to withstand thermal and radiation exposure, the coating could also be used for **ex situ prevention**. The Gentoo™ coating material was developed by LUNA on a contract from the Department of Defense Small Business Innovation Research (SBIR) program and it was licensed to Ultra Tech International to make it commercially available. Gentoo™ has been applied to several different applications, including applications with DOE, DOD, and NASA. Gentoo™ can be modified to tune the properties toward use on SNF canisters. Gentoo™ has been used in other corrosion prevention applications and has demonstrated effective corrosion resistance. Specifically, it has been applied to Duplex 2205 SS and shown to increase the pitting potential and decrease the anodic reaction by two orders of magnitude.

### 3.5 CLADCO™

Like Gentoo™, the CLADCO™ coating proposed by FC, is an organic/inorganic hybrid coating. CLADCO™ comprises a hybrid quasi-ceramic coating consisting of a single component (1K) polyurethane polymer linker and a silica-based sol-gel ceramic component. The 1K polyurethane polymer provides a flexible hydrocarbon layer that, according to FC, adheres strongly to the metal surface and improves mechanical stability—actual values have yet to be determined. The ceramic component offers great chemical and thermal stability. This coating can be seen in Figure 9 applied to a 6" x 6" SS coupon. While polyurethane alone was not considered a viable option in the FY20 report due to issues of thermal expansion, it is unknown if those issues are alleviated when polyurethane is combined with a ceramic coating in the hybrid CLADCO™ (or Gentoo™). As mentioned previously, the hybridization of sol-gel coatings through the incorporation of organic compounds can result in superior corrosion resistance through strong chemical bonds between organic and inorganic components and improved chemical physical stability; however, the resulting material could potentially be susceptible to radiation or thermal damage [1]. The inorganic ceramic component is radiation resistant, but the organic component has the potential to undergo radiolysis [37]. The amount of radiation required to cause damage can vary significantly with different organic components, therefore specific research into CLADCO™ is required to determine appropriateness for use on SNF canisters.

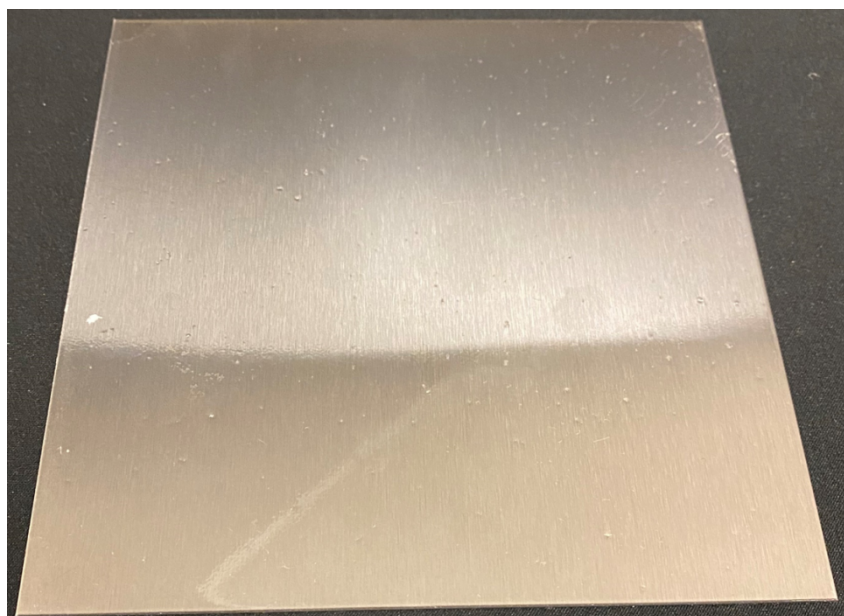


Figure 9. A coupon coated with CLADCO™ provided by FC.

The proposed FC solution to address potential CISCC in SNF canisters is to use CLADCO™, which is commercially available, as a low viscosity liquid that can be applied (via spraying or brushing) to a wide array of substrates and dries under ambient conditions – without the need for external heating. The resulting coating is chemically inert and transparent with a thickness that can be varied to extend the lifetime of the coating. The coating also exhibits a low surface energy and strong adhesion to SS. According to FC, CLADCO™ adheres strongly to the surface and condenses to create a physical barrier against corrosion. FC has previously worked with several academic and commercial partners as well as the DOD through the US Navy and Army to develop coating technologies to address specific purposes.



## 4. EXPERIMENTAL TEST PLAN

The purpose of this experimental plan is to evaluate the properties of the candidate coatings and determine their viability as a solution to prevent, mitigate, and repair potential CISCC on SNF DSCs. Physical properties, such as the adhesion strength and coating hardness, will be measured to determine the expected durability of the coating under realistic conditions. The proposed electrochemical measurements are designed to determine the relative corrosion mitigation performance of the coatings, and to gain an understanding of their performance in short and long-term application. A comprehensive understanding of the candidate coatings is required to ensure that use of the coating will not introduce any additional problems to the SNF DSC surfaces.

Some possible ways that an applied coating could do additional harm include: premature failure; enhancement of cathodic reactions (including hydrogen evolution) on the surface and those that pose a risk for hydrogen embrittlement; generation of crevices or occluded environments that can exacerbate corrosion; generation of particles that can cause obstruction of passive ventilation features in the system; or the requirement of frequent maintenance or repair (thereby increasing handling of the storage components). Therefore these considerations must be taken into account in determining a coatings viability. While the goal of a corrosion mitigation coating is to stop corrosion from occurring, in this application it must first demonstrate that it “does no harm”. Exhaustive studies of the physical and chemical properties of the candidate coatings in *Phase 1* of the MOU are aimed at ensuring that future resources are allocated on coatings that fulfill this basic principle. The remainder of this section describes specific methods that will be used in *Phase 1* (Phases described in detail in Section 2.1), which includes base line measurement performed in FY21 and coating evaluation analyses beginning in FY22. The remaining *Phases 2 & 3* will be addressed in subsequent reports.

Under the MOU, SNL will send out SS coupons to each of the participating industry partners. The coupons will be coated and then sent back to SNL for experimental testing and analysis.

### 4.1 Coating CharacterizationError! Reference source not found.

Coatings will be inspected visually by SNL upon receipt to document the color, texture, and other observations of each coating. A documented visual inspection is because the coating may change throughout the course of the planned tests or with increased aging exposure. Color changes can indicate at the most basic level that degradation has begun (due to thermal, chemical or other environmental degradation mechanisms). Texture and surface roughness are important factors because they may impact how salts or dusts accumulate on the surface, with smoother surfaces retaining fewer particulates than a rougher surface. The surface texture of the coating may be characterized using a scanning white light interferometer over a large area to capture a representative surface roughness value. Comparisons of roughness throughout the test phases will help develop an understanding if any dissolution or non-uniform attack of the coating has occurred. Other microscopic (e.g. scanning electron microscope (SEM)) and spectroscopic (e.g. FTIR and Raman) will be performed to evaluate the structure at a micron-level scale as well as identify major contribution functional groups of the as received samples.

Following physical, chemical, and electrochemical testing (described in Section 4.2 and Section 4.3, respectively) a focused ion beam (FIB) may be used to remove a small portion of the coating to enable SEM of the cross-section of the coating/substrate interface. Energy dispersive X-ray spectroscopy (EDS) will be performed on the cross-section to determine the distribution of elemental constituents.

### 4.2 Physical Tests

#### 4.2.1 Adhesion Tests

The adhesion strength of a coating to a substrate is most effectively evaluated by controlled physical destruction of the coating. *ASTM D4541-17* describes a direct approach to characterize the adhesion of a

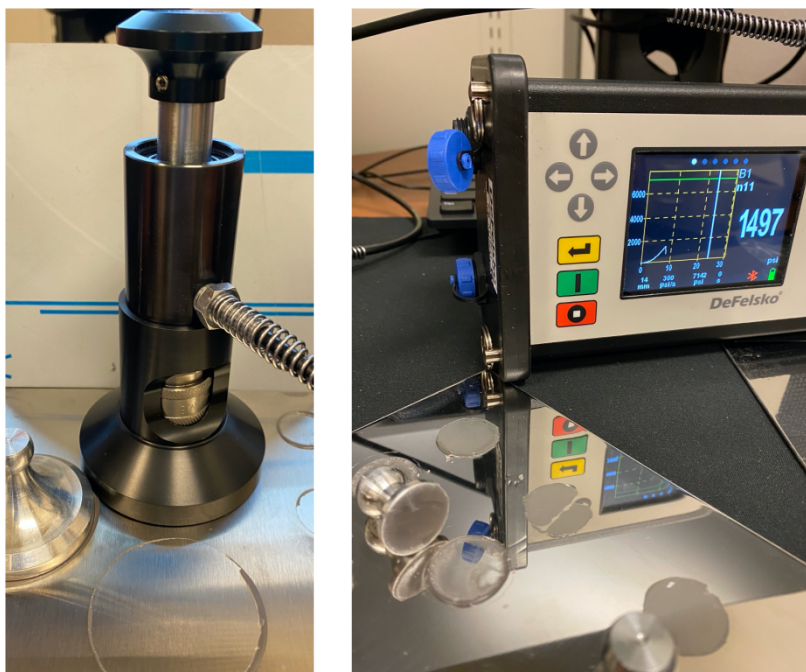
coating [38]. This test quantifies the load required to remove a coating from a substrate by measuring the “pull-off” force. To test adhesion using this method, epoxy (ResinLab ® EP11HT gray 2-part epoxy [39]) is applied to the cleaned bonding surface of a test stud which is pressed firmly onto the surface of the coating. An epoxy dispensing gun with a mixing nozzle is used to precisely control the mixing ratio of the epoxy. The epoxy is dried for at least 24 hours under ambient conditions to ensure a solid bond. A portable, automatic adhesion tester (Figure 10) is then attached to the stud and a uniform, gradual load is applied to the stud perpendicular to the coating surface. The test is complete when the stud is removed from the surface, resulting either from epoxy failure (maximum reported adhesion value is approximately 1800 PSI), coating failure, or when the maximum load of the tester is reached (3500 PSI for 20 mm test studs [40]). The portable tester uses an internal transducer to measure the load that it applies on the stud in real-time. Typical loading rates are  $\sim 1\text{ MPa/s}$  ( $150\text{ psi/s}$ ) which results in a test time of less than 100 seconds. The coating can be scored with a circular cutter if the coating is thick to ensure that only the area inside the stud footprint is tested though the possibility of introducing microcracks exists. Variation in measurements are typically on the order of 17.5% - 23% as characterized in the *ASTM D4541-17* standard [38]. All coatings will be tested using this method, with at least two replicates per coupon.



**Figure 10. Automatic adhesion tester (left) and cross-sectional schematic of pull-off mechanism (right) [38].**

#### **4.2.1.1 Baseline Adhesion Measurements of the Epoxy to the Base Metal**

Initial baseline measurements testing the adhesion of the standard ASTM epoxy to the base metal material were performed to quantify a maximum value for the pull-off test. This test provided an estimate for the maximum force required to remove the stud from the metal surface after being epoxied to the surface. In these tests, the stud was adhered to the surface using the 2-part epoxy either mixed by hand or using an epoxy mixing/dispensing gun onto both the rough ( $R_a = 0.359\text{ }\mu\text{m}$ ) and smooth ( $R_a = 0.026\text{ }\mu\text{m}$ ) sides of the SS coupon – surface roughness measurements are shown in Figure 17 and described in Section 4.4. The experimental set up is shown in Figure 11.

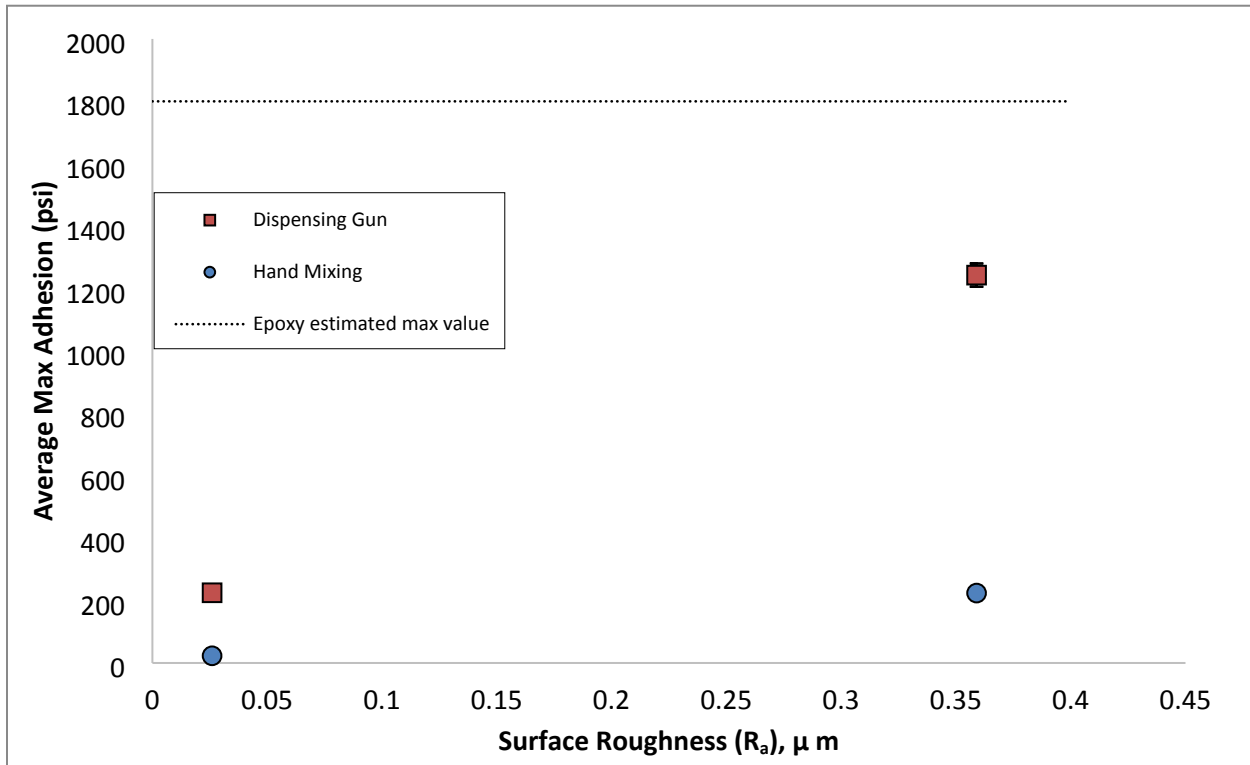


**Figure 11. Experimental test of the baseline measurement to determine the strength of adhesion of the epoxy to the base metal. These measurements determine the maximum adhesion value that can be measured.**

The results show significant variation both as a function of the surface roughness and as a function of how the 2-part epoxy was mixed (Table 3). It is clear from Figure 12 that the measured max adhesion values are dependent upon the surface roughness and the epoxy mixing method. From this result, SNL will require the use of the dispensing gun for mounting the stud with the epoxy to the surface of the coated or bare coupon. Furthermore, additional work will be required to determine the relationship between surface roughness and the epoxy adhesion strength. Since it is expected that the coatings will have different surface roughness, a robust understanding of the theoretical maximum strength of the epoxy as a function of surface roughness is required. Although the epoxy may fail under different forces as a function of surface roughness, it will be visually obvious to identify whether the epoxy fail or if the coating failed during the test.

**Table 3. Baseline adhesion values of epoxy on the base metal.**

Sample		Average Max Adhesion (psi)
Rough Surface ( $R_a = 0.359 \mu\text{m}$ )	Dispensing Gun	225
	Manual Hand Mixing	23
Smooth Surface ( $R_a = 0.026 \mu\text{m}$ )	Dispensing Gun	1243
	Manual Hand Mixing	224



**Figure 12. Measured adhesion values for the epoxy on the base metal for rough and smooth surface when the epoxy is applied via dispensing gun or hand mixing. The maximum expected pull-off strength of the adhesive is 1800 PSI.**

#### 4.2.2 Scratch Tests

Scratch tests will be performed under the procedure provided in the ASTM Standard *D7027-13 Standard Test Method for Evaluation of Scratch Resistance of Polymeric Coatings and Plastics Using an Instrumented Scratch Machine* [41]. This standard describes a destructive physical test with which the mechanical robustness of a coating can be compared directly with other coatings. These measurements are typically performed using a commercially available scratch tester (Figure 13 and Figure 14). Scratch testing is a common practice and is used to characterize all types of coatings in many different industrial applications (research, quality control, production, etc.). Typically, a diamond-tipped stylus (radius and geometry can be varied by exchanging tips) is progressively loaded (from low load to high load) while being rastered uni-directionally (once or repeatedly) across the surface of the test specimen. The instrument measures the normal load (on the diamond tip), the lateral load due to friction, and any acoustic emissions that may result from sliding. Acoustic emissions data will not be used for this experimental test plan. The resulting scratch on the coupon is optically evaluated using a microscope to find the onset of different failure mechanisms such as cracking, cohesive failure, and complete adhesive failure (Figure 15). The distance between the start of the scratch and the onset of these failure mechanisms can be used to determine the normal and lateral forces exerted on the coupon at the observed locations. Depending on the optical properties of the coating, it may be necessary to further evaluate the features in the scratch using an SEM to determine the precise location of the onset of failure. The forces at these onset locations are compared across coating types, batches, lots or variants to determine performance.

Scratch testing can be used to *infer* some physical properties (e.g. adhesion) but is typically used as a comparative method to evaluate scratch performance of a series of coatings. Another property acquired during the scratch testing is a quantification of the dynamic friction coefficient since the lateral sliding force is measured during the test. Since scratch testing has been shown to be operator-dependent and may have issues with repeatability, it will only be used to collect a coatings baseline scratch performance and compare those results to a coatings scratch performance after exposure to cyclic atmospheric conditions to gain a better understanding of the impacts of environmental exposure on the coating. Since many of the coatings to be tested are polymeric, it may not be possible to identify the correct failure mechanism from the scratch. Ductile coatings may not fail correctly for characterization in a shear-mode scratch test, but this will be noted and identified when applicable.

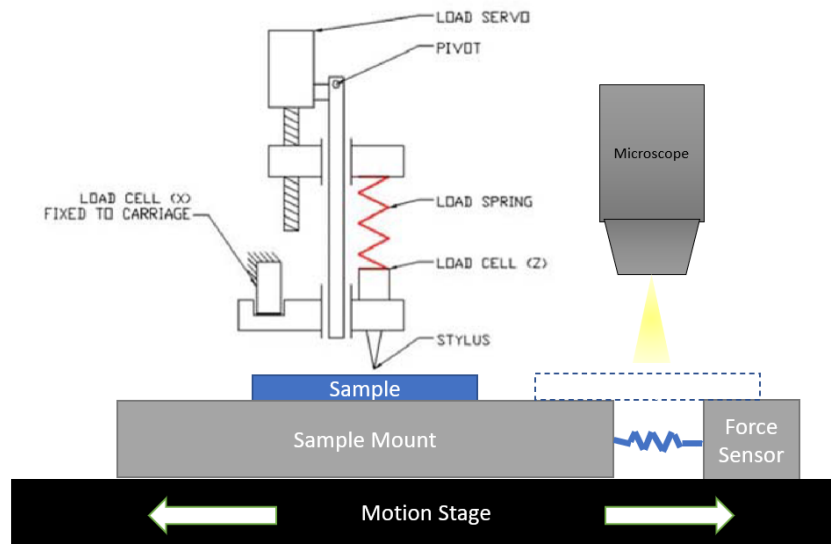


Figure 13. Basic layout of a scratch tester (adapted from ASTM D7027-13 [41])

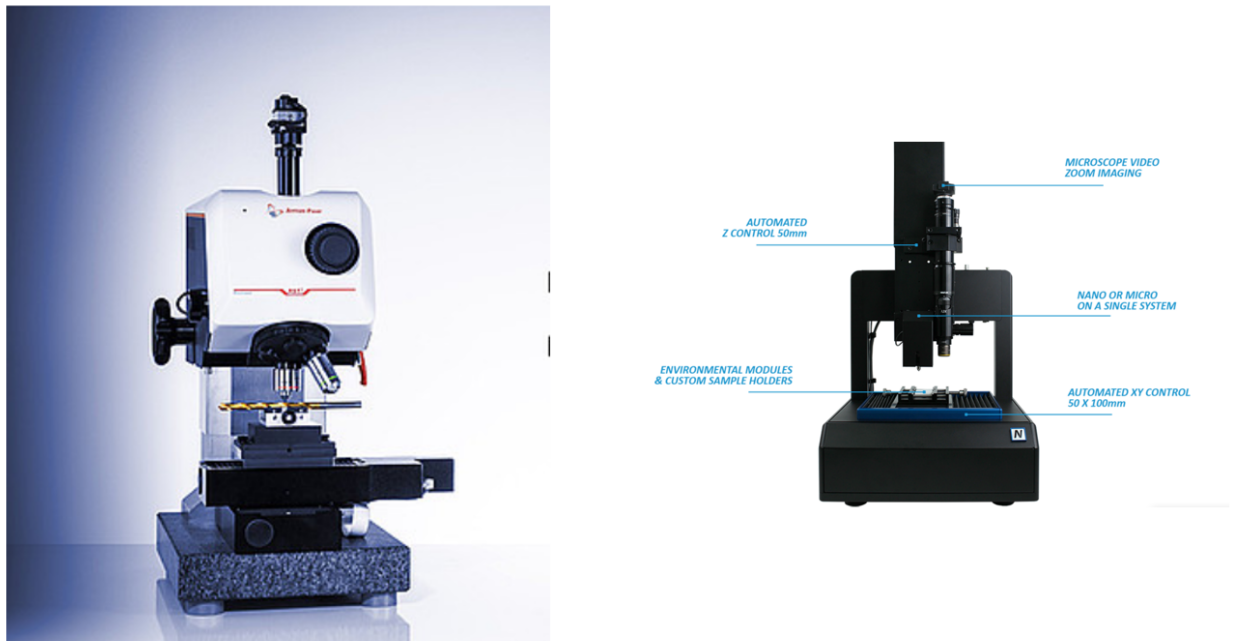


Figure 14. Commercial scratch testers [42, 43].

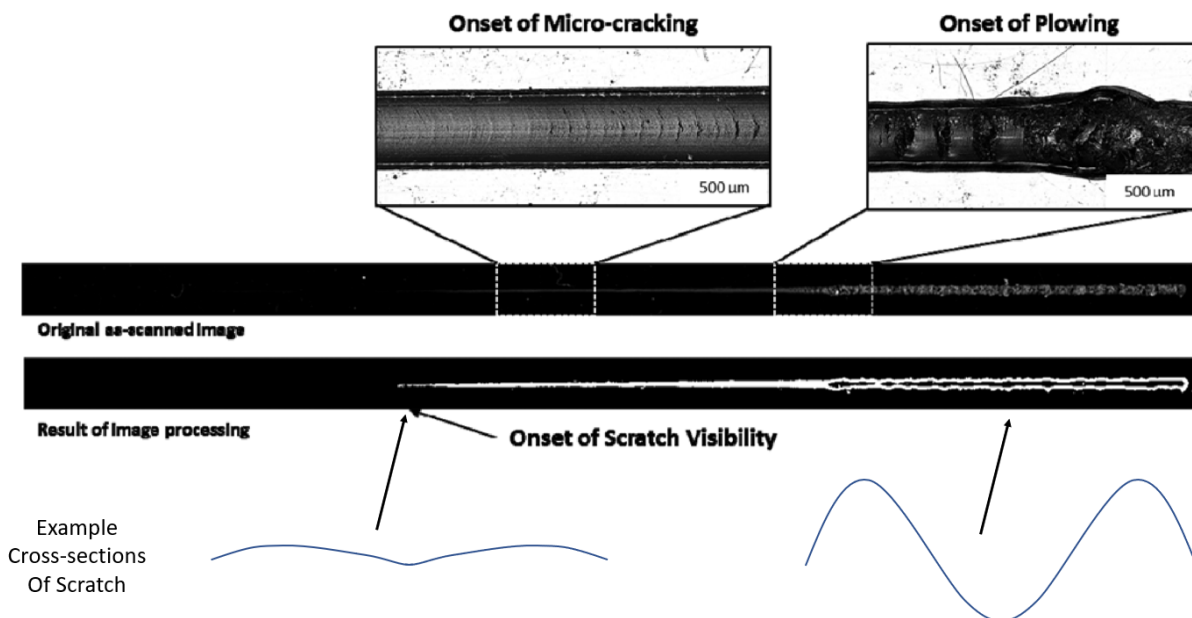


Figure 15. Scratch test appearance and depiction of onset of failure mechanism (adapted from ASTM D7027-13 [41]).



### **4.2.3 Hardness and Thickness Tests**

Hardness and thickness are physical parameters of a coating which play a huge role in determining the durability of the coating and its ability to survive transient mechanical events – such as movement and transportation.

Hardness is a measure of a coating's ability to resist deformation. Factors such as a materials' elastic modulus, ductility, ultimate tensile strength, and viscoelasticity (for polymers) can all contribute to the hardness of a material. To characterize hardness, a controlled-geometry indenter, such as a ball or chiseled tip, is pressed into the sample with a known force. The penetration of the indenter into the sample is either measured directly or indirectly (via a post-test optical measurement of indent size). The geometry of the indenter, the force applied, and the resultant penetration are usually expressed in a dimensionless hardness factor which is test-method dependent. Common test methods include Vickers, Knoop, Rockwell and Brinell [44, 45]. Hardness measurements can be taken individually or can be used to map a large area with an array of measurements. Hardness measurements on thin coatings can be complicated as the applied forces are large and the penetrations necessary to make the measurement may be greater than the coating thickness.

Thickness can be characterized using a variety of methods including crater grinding [46], capacitive gauges [47], and ultrasonic gauges [48]. Thickness of the candidate coating is an important parameter in determining the expected lifetime and performance of a coating. A coating with a low permeability but also a small thickness may not prevent corrosion for long enough, where as a coating with a higher permeability but a higher thickness may have sufficient life expectancy. Thicker coatings are also more susceptible to delamination as the residual and thermal forces increase as the material volume increases. Thinner coatings can be more resistant to delamination, but small defects, which are hard to prevent, can compromise the coating integrity. An in-depth understanding of the relationship between the coating thickness and coating properties will help identify which properties scale linearly with coating thickness. Once this relationship is understood, the coatings will be compared to one another via the normalized coating properties.

Capacitive gauges seem to be the most readily available commercial method to evaluate coating thickness, but the range of thicknesses they can measure accurately is limited. Capacitive gauges also rely on knowing the electronic properties of the coating, which may not always be known. Ultrasonic gauges are also limited in the range of thickness measurement and do not have sufficient accuracy for measuring thin coatings. Crater grinding is a viable method, but the equipment is expensive, and the measurement consumes a large area on the sample. Coating thickness can also be determined by eroding the coating to the substrate using a focused ion beam. Once the coating is fully eroded, an SEM can be used to measure the cross-sectional thickness of the coating in the eroded area. Unfortunately, this method is only valid on extremely smooth coatings and substrates since surface roughness will play a significant role in the measurement of thickness in the eroded area. Also, since the eroded area is quite small the sampling length is also limited and may not represent the overall thickness accurately. A stylus or optical profilometer may also be used, but this requires a masked or uncoated area to be left on each specimen. This is not the best approach because an uncoated area may present opportunities for corrosion to nucleate during the proposed corrosion exposures which would unfairly bias the results. Also, the coating may be thicker around the mask edges which may skew the result.

Nanoindentation is a method that uses nano-scale forces to measure the properties of a material. Nanoindentation can be used to measure the hardness of a coating on a hard substrate while correcting for substrate effects. More importantly, nanoindentation can also collect hardness versus indentation depth. Since the hardness properties of the substrate are known or can be measured prior to coating, nanoindentation can also be used to measure the thickness of the coating [49]. Nanoindentation will be performed as the main method of characterizing the thickness and hardness of the candidate coatings. Hardness may also be measured using other methods if thickness is known.

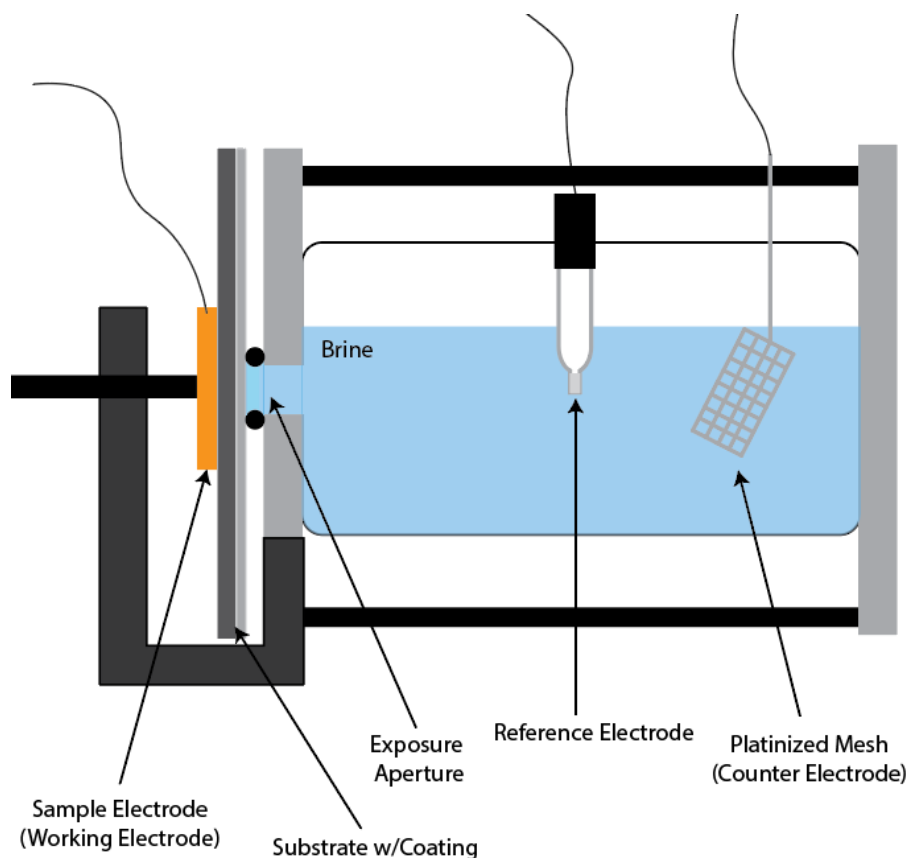
## 4.3 Chemical and Electrochemical Tests

The electrochemical performance of the coating is the primary factor to evaluate for coatings performance, as reduction in corrosion will reduce the potential for CISCC. Poor electrochemical properties will lead to rapid ingress of corrosive species to the substrate surface and result in continued or even accelerated corrosive attack. Prevention of corrosive attack in the coatings selected here will occur primarily through prevention of ingress of electrolyte to the canister surface. Flaws or weaknesses in the coating such as pores, cracks, or pits may cause rapid breakdown of coating performance and/or direct corrosion of the substrate. Coatings that are highly permeable allow the rapid exchange of ionic species between the anode and the cathode through the intact coating. Rapid ion exchange could lead to rapid corrosion. Electrochemical performance is characterized using a wide variety of techniques, many of which result in a comprehensive understanding of corrosion performance in application specific environments.

### 4.3.1 Full Immersion Testing

Long-term full immersion exposures in electrolytes of interest will be carried out to determine general coating resistance to corrosion and potential degradation over time. Electrolytes of interest include 0.6 M NaCl (for benchmarking versus literature/historical data) and artificial seawater solutions based on conditions relevant to canister exposure (for example equivalent seawater mixtures for a 40 % RH brine). Full immersion exposures will be periodically interrupted with electrochemical impedance spectroscopy (EIS) measurements following the ASTM-STP-1188-EIS procedure to acquire quantitative measurements of coatings degradation, such as coating delamination. EIS will be taken at open circuit potential ( $E_{OCP}$ ), in an electrochemical cell similar to that depicted in Figure 16. Electrochemical measurements using this cell, equipped with a Ag/AgCl reference electrode and Pt counter electrode, will be carried out with a BioLogic potentiostat. EIS will be acquired in sine sweep mode from 100 kHz to 0.01 Hz with 6 points per decade. Coated samples will be scanned with an alternating current (AC) voltage of 50 to 80 mV to overcome the potential drop in the coating and minimize noise. An example test setup for measurement is as follows: Initial EIS measurement in 0.6 M ambiently aerated NaCl followed by a 1 h  $E_{OCP}$  measurement, then a second EIS cycle, followed by a 6 h  $E_{OCP}$  measurement, a third EIS measurement and so on. This same periodic EIS/ $E_{OCP}$  measurement will be carried out over extended time intervals such as 1 h, 6 h, 12 h, 24 h, 2 days, 7 days, 28 days. The EIS measurements will then be fit to an equivalent circuit and parameters such as pore resistance, the delamination parameter, and the breakdown frequencies. The extrapolation of these parameters and how they vary with time will provide indication of the coating's corrosion resistance over time and exposure environment.





**Figure 16. Schematic of a typical corrosion flat cell**

#### 4.3.2 Relevant Atmospheric Environments

In addition to corrosion evaluation in full immersion environments, coatings degradation will be quantified under atmospheric exposures of interest. A similar experimental setup will be applied to expose samples as those described for large scale pitting analysis in the FY20 Milestone report [11] (Section 2.2.2.1 Arkansas Nuclear 1 for Cyclic Exposure conditions and 3.1.1 for static exposure conditions). For corrosion evaluation herein, coated samples will be exposed to the atmospheric environment of interest and then removed from the exposure for EIS measurements. The first set of samples will be exposed for a short period of time (hours to weeks) followed by EIS measurements and other destructive tests. The second set of samples will be exposed for a long period of time (weeks to months) followed by EIS measurements and other destructive tests. Time scales of atmospheric exposure will be similar to those selected above for full immersion exposures. EIS measurements will be performed in a three electrode electrochemical cell (Figure 16) in the solution comparable to that of the exposure conditions (i.e. 40% equivalent RH seawater solution), and at the open circuit with the same parameters as those described for full immersion measurements. Additionally, surface imaging and microscopy will be carried out to qualitatively evaluate the coating breakdown over time. Extraction of pertinent EIS parameters (e.g. resistance) will provide a quantitative measurement of coating degradation. Additionally, adhesion or permeation testing could be applied to gain measurements of the properties post exposure (on areas of the coupons not consumed for EIS measurements as EIS measurements are considered destructive).

### 4.3.3 Outgassing

Outgassed species from potential coatings could enhance the corrosion rate of a system by introduction of accelerating species such as sulfur or chlorides. Outgassing is the result of a partial pressure differential between the ambient environment (or test environment) and low vapor pressure components in a solid material (such as solvents). This is especially important at elevated temperatures where diffusion of volatile species is accelerated. Since, the primary goal of a corrosion mitigation coating in SNF canisters is to “do no harm”, a corrosion mitigation coating that outgasses a corrosive species may cause more harm than it prevents and therefore should not be used. Thus, measuring the outgassing performance of the candidate coatings is important in determining the suitability of a coating to be used in this application.

Outgassing is measured in a vacuum environment at elevated temperatures. ASTM E595-15 [50] describes a standard practice for measuring the total amount of outgassed species from a material. The method involves exposing the sample to a humid environment for 24 hours to saturate the sample with water. This is important in determining the water uptake of the coating. Next, the sample is mounted on a heating plate inside a vacuum chamber which is then evacuated to  $1 \times 10^{-5}$  torr or better. The sample is heated to 125°C and left for 24 hours to outgas into the vacuum chamber. A second section of the vacuum chamber contains a chromium plated chilled condensing plate which condenses volatile gases that are outgassed from the sample. After 24 hours, the sample is cooled to room temperature, the vacuum chamber is repressurized with dry inert gas, and the sample is removed. The condensing plate is weighed to determine the mass of the condensate – this mass is quantified as the collected volatile condensable materials (CVCm). The sample is also weighed to determine the amount of mass that was lost from the sample – this is the total mass loss (TML). According to the standard, a material with a low CVCm ( $<0.1\%$ ) and a low TML ( $<1.0\%$ ) is defined as a low outgassing material and would be suitable for use in spacecraft. Unfortunately, this method does not describe the characterization of the outgassed species, which is of critical importance to this effort. A material which may be labeled as low outgassing according to ASTM E595-15 may still outgas low concentrations of “bad actors”. Additional analyses can be performed at SNL to determine the identity of the degassed species by spectroscopic or chromatographic methods. Following a quantified measurement of the CVCm and TML, samples will be qualitatively analyzed to determine which species are being outgassed as a function of temperature. SNL’s Materials Reliability Department specializes in making high sensitivity measurements of outgassed components to identify trace-level chemicals which could cause damage in hermetically sealed systems. They have developed customized methods to characterize both the short-term outgassing behavior of a material and long-term aging-related chemistry that might evolve over time. In this case, the sample is heated in a vacuum environment in the presence of a residual gas analyzer (RGA) or mass spectrometer (MS). As the different volatile species escape from the sample into the vacuum environment it is detected by the RGA or MS and logged as an atomic mass. These atomic masses can be analyzed and compared to standards which can indicate exactly what species evolved from the sample and in what concentration. When logged continuously, it is possible to determine how the concentration of these species evolves over time. This procedure will be performed on a pristine section of all candidate coatings after all other tests have been completed.

## 4.4 Testing Protocol

Coatings will be applied to 3” by 6” square UNS30400 SS substrates with a thickness of 0.065”. The substrates have been polished on one side ( $0.026 \pm 0.002$  micron  $R_a$ ) and ground on the other ( $0.359 \pm 0.011$  micron  $R_a$ ). See Figure 17 for roughness measurements.

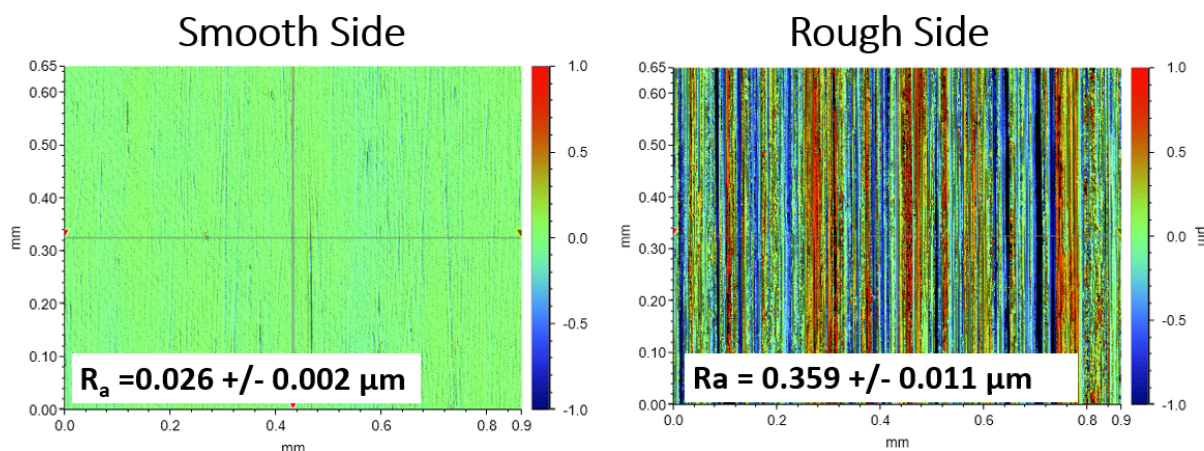


Figure 17. Optical profilometry results on uncoated substrates

Collaborating industry partners will be supplied with enough coupons to perform the proposed tests shown in Figure 18 on one side of the coupon (minimum of eight coupons for the smooth side, and eight coupons for the ground side). One side on each coupon will be left uncoated to allow for connection of electrodes used in the corrosion tests. This will allow the characterization of the physical and electrochemical properties of the same coating on two different surface finishes. Surface finish plays an important role in adhesion of the coating.

The experiments described in Sections **Error! Reference source not found.** and **Error! Reference source not found.** and shown in Figure 19 will be conducted over the surface of the coated coupon in a specific order (Figure 20). Recognizing that certain tests, such as the adhesion, scratch, EIS, hardness/thickness, are destructive to the coating surface; while others, such as optical imaging, microscopy, and spectroscopy are non-destructive tests and therefore will be conducted first. Each of the four coupons shown in Figure 18 will be subjected to different conditions to develop an understanding of the coating performance in a few different scenarios.

Baseline measurements will be conducted on Coupon Set 1 (Figure 18). Coupons are exposed in sets of two 3"x6" samples. EIS, hardness, thickness, scratch tests and optical characterization tests will be performed on the first coupon in the set. Adhesion tests are performed on the second coupon in the set to prevent damage to areas where other tests need to be performed. After all tests on each coupon set have been completed, outgassing studies will be conducted on an undamaged piece. Duplicate tests are performed on each coupon regardless of exposure to improve statistical significance of the results.

Coupon Sets 2 and 3 (Figure 18) will be exposed to certain cyclic atmospheric conditions designed to mimic typical storage conditions seen at the surface of SNF canisters. These conditions include full immersion and atmospheric exposure conditions, which are described in Section 4.3.1 and Section 4.3.2, respectively. After the first time interval, Coupon Set 2 will be removed from these conditions, cleaned, and tested using the same protocol as Coupon Set 1. After the second time interval, Coupon Set 3 will be removed from the exposure, cleaned, and tested using the same protocol as Coupon Set 1.

Two additional coupons will be used as a control and as an EIS witness coupon to observe the impact of immersed conditions over time. One coupon will be stored in a desiccator, as a control, for the entirety of the experimental period. The EIS witness coupon will be mounted in a corrosion flat cell (Figure 16) and exposed to immersed conditions over a small area (~1 cm<sup>2</sup>) in the middle of the coupon. Electro-impedance Spectroscopy (EIS) will be performed at regular intervals of hours or days between measurements. The immersed conditions on this small area will be maintained for 12-18 months to assess the long-term coating performance. This continuous measurement during the exposure will allow the

direct measurement of the coating degradation over time. These changes may occur in the form of porosity, diffusion of ionic species from the brine into the coating, or dissolution/thinning of the coating. This test procedure is described in 4.3.1 .

The control specimen will be stored in the desiccator for the same amount of time as the immersed sample will be exposed. The adhesion and scratch data from the control specimen (collected at the end of the exposure tests) will be compared to the same measurements on Coupon 1 and to the non-exposed areas of the EIS coupon in Figure 18 (which was not stored in a desiccator). Differences between Coupon 1 and the control coupon may indicate that the coupon is changing over time even in inert storage. Differences seen between Coupon 1 and the non-exposed area of the EIS coupon may indicate a sensitivity to RH, oxygen, or other conditions present in the ambient environment.

The test protocol will be performed the same way on both smooth and rough coated coupons.

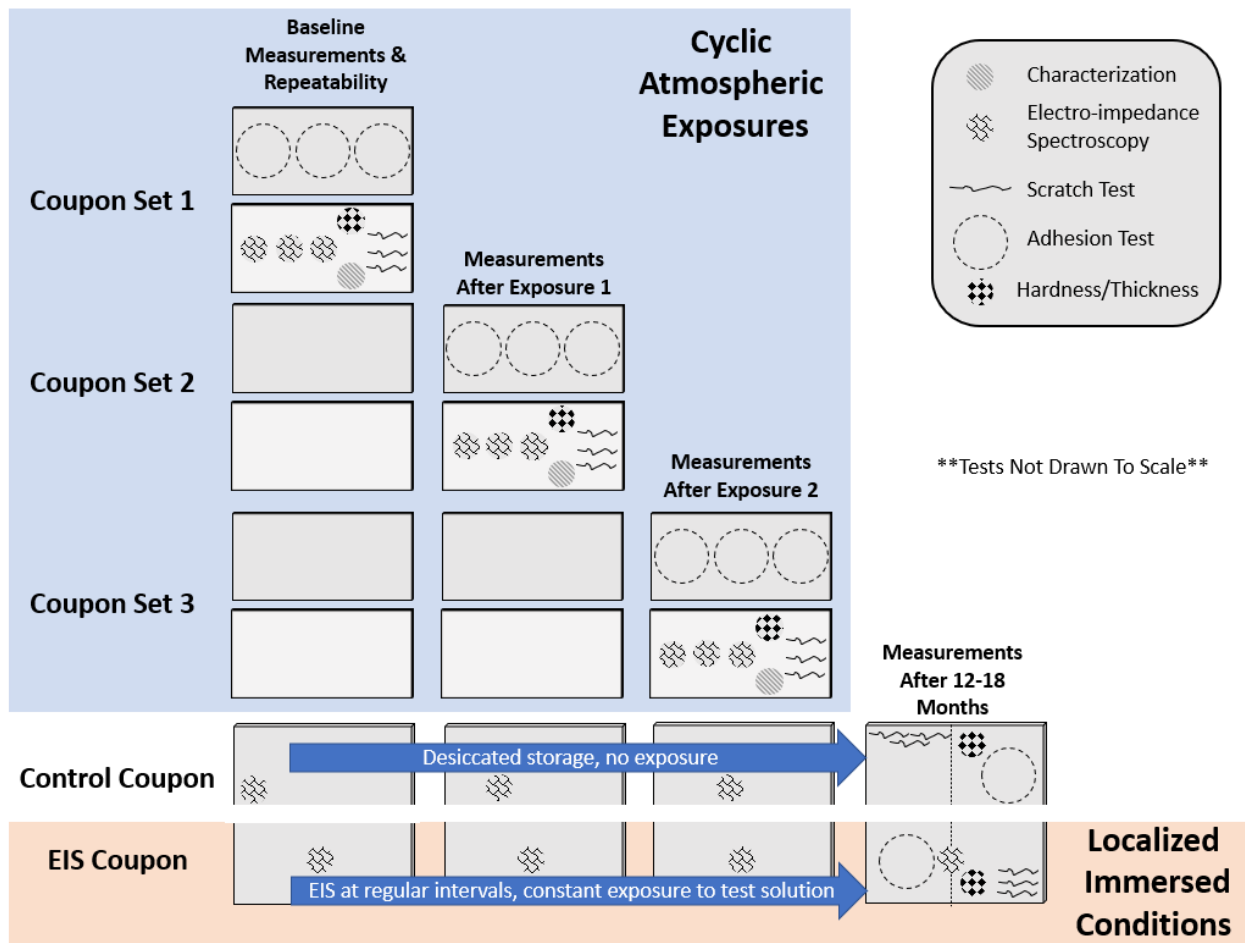


Figure 18. Test matrix of coated coupons (testing locations shown but are not to scale) to be performed on smooth and ground sides of coupons.

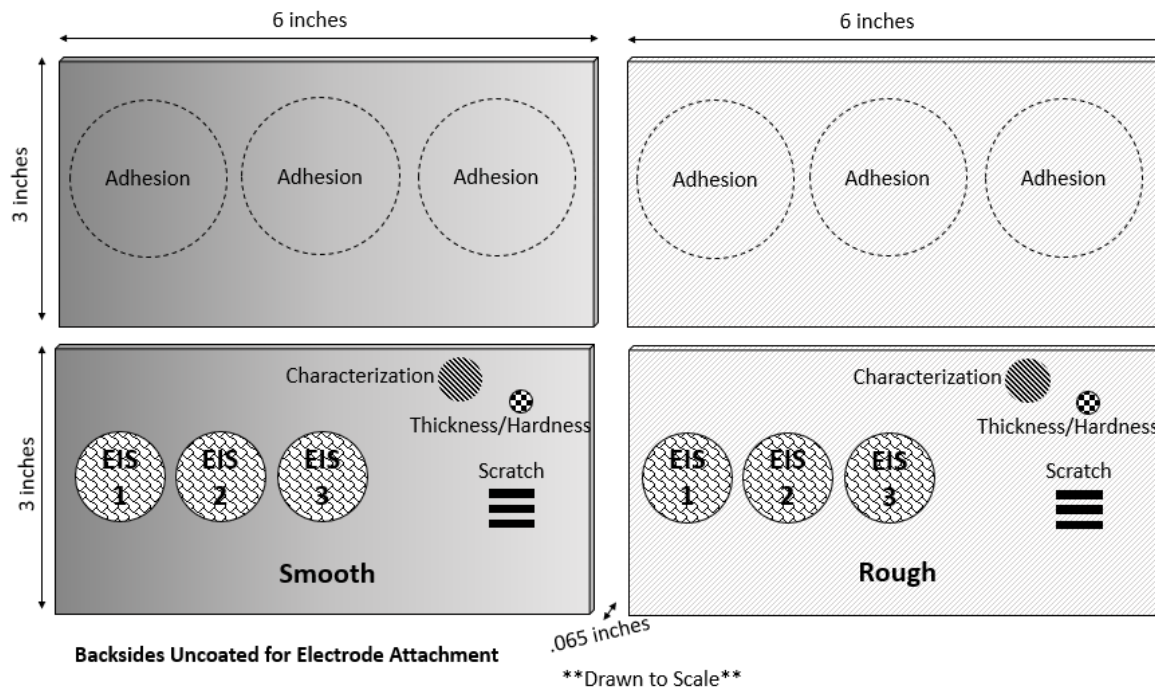


Figure 19. Example of individual tests on a coated coupon set (testing locations shown to scale – but size exaggerated by 2x) from Figure 18.

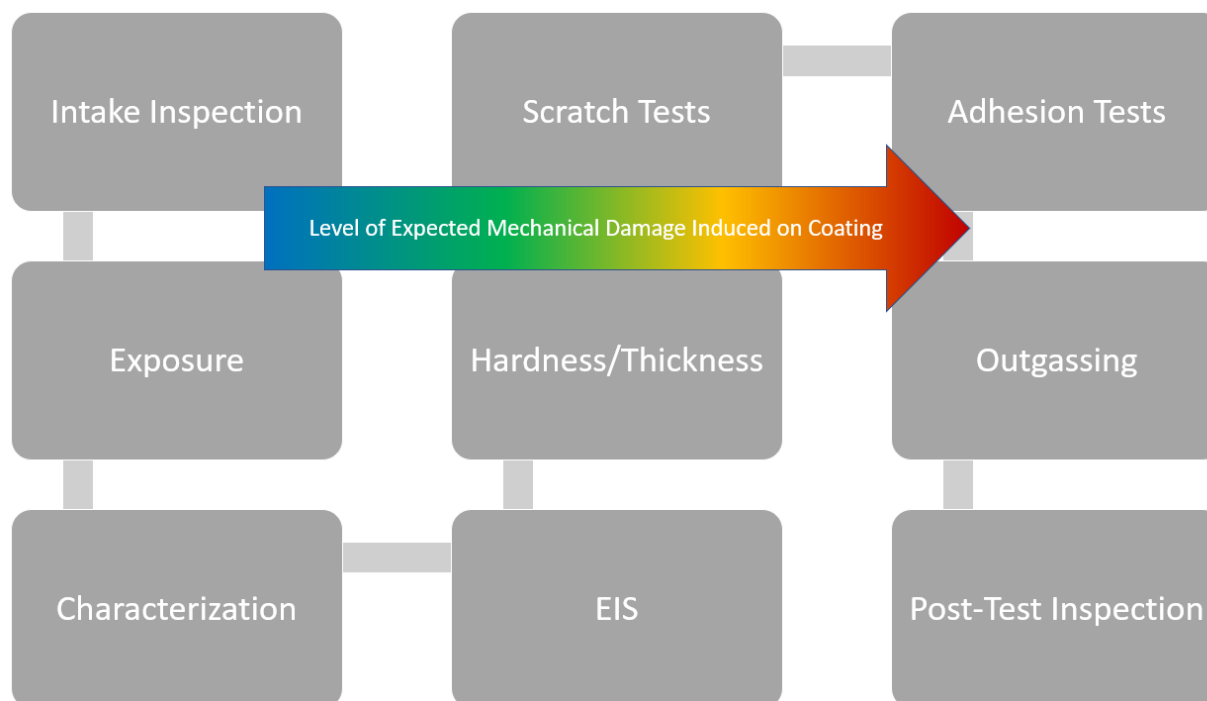


Figure 20. Test progression for coupons 1, 2 and 3 in Figure 18.



## 4.6 Anticipated Work Outcomes for FY22

As mentioned in previous sections, an intimate understanding of the chemical and physical properties of the candidate coatings is desired. A candidate coating must be fully characterized in order to ensure a safe and effective implementation on SNF canisters. A coating which may cause additional material concerns cannot be introduced into the SNF storage system. Further, quantification of corrosion mitigation performance is another important metric by which all coatings will be compared. If a coating does not provide adequate corrosion protection, it does not meet the basic function of the coating and will not be considered for further research. Finally, an understanding of the manufacturing, cleaning and application processes for the candidate coatings is also sought. Part of the initial phase of the MOU relationship with coating manufacturers was to solicit responses to a survey requesting information regarding several factors in a coating's feasibility not relating to performance. These include:

- **Scalability:** A coating which cannot be applied at scale cannot be considered for this application because of the large volume of material required. Over 3,158 canisters are already in use and in 2020 alone, 222 new canisters were filled [17]. Thus, the solution must be scalable for high volume production. This also is dependent on the end use of the coating. If the coating is meant to be used as a patch repair, rather than a conformal coating over the entire canister, the production volume might be relatively low. In addition to the number of canisters that need to be coated, the scalability must also account for the coating to be able to coat large items like the SNF DSCs.
- **Required surface preparation methods:** Surface preparation methods are also relevant to the impact of the coating process on the canister storage environment. Introducing acids or other corrosive chemicals in the storage environment may be extremely detrimental to the performance or integrity of the surrounding structures in the SNF storage system. Introduction of large quantities of moisture could cause corrosion conditions which were not initially present. Surface abrasion is a common method used to roughen and remove oxide films from the substrate surface before coating. Excessive abrasion could introduce surface stresses and scratches which, if left uncoated, could initiate crevice corrosion.
- **Application method:** Application methods that require complicated or multi-step treatment processes may not be feasible due to industry reluctance to handle the canisters excessively or to conduct repair processes over an extended period of time. Further, **in-situ repair** application methods are limited to tools that can be inserted through the passive air inlets. These access limitations restrict the size and capabilities of equipment that can be inserted into the gap between the overpack and the canister surface.
- **Application/curing parameters:** Application and curing parameters are conditions that must be present to achieve maximum coating performance. Coatings which require elevated cure temperatures may not be feasible solutions for **ex-situ** and **in-situ repair** scenarios because the introduction of additional energy sources into the SNF storage system may cause increased risk of rupture.
- **Contaminants in the coating or its precursors:** Contaminants that may be present in the coating or its precursors could have a large impact on the overall corrosion mitigation performance of the coating. Chlorides as contaminants would likely cause corrosive attack at the interface between the coating and the substrate. Outgassing measurements described in Section 4.3.3 should reveal these contaminants if they are volatile, while coating characterization described in Section 4.1 should reveal contaminants that are bound within the coating or are concentrated at the coating/substrate interface.

Consideration of these factors, combined with the relative performance of the coatings in the proposed tests, will allow development of a thorough understanding of SNF canister coating options. A few additional considerations include coating costs, which may be an important facet of scalability;

also potential supply chain vulnerabilities may pose challenges for a coating to be a reliable solution as the demand may continue to grow for decades.

Phase 1 testing should reveal which coatings should be targeted to specific application scenarios and what improvements must be made to address specific needs for eventual use on SNF DSCs. Candidate coatings may require optimization of different parameters described above. For example, some coatings may require optimization of the curing process while others may require optimization of surface preparation methods. Coatings with a high curing temperature may be ideal for use on new canisters since they can be subjected to curing temperatures during production. Some coatings that have poor adhesion on dirty surfaces, but strong adhesion on pristine surfaces may be better applied on new canisters or on canisters which can be safely removed from the concrete overpack and thoroughly cleaned. Coatings which enable the use of a robotic crawler during application but have a shorter service lifetime may be better suited to **in situ repair** application scenarios. Initial measurements in Phase 1 will also facilitate the development of detailed test plans for Phase 2 coating optimization activities and more rigorous thermal and radiation testing.

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## **5. CONCLUSIONS**

This report summarizes the current activities in FY21 related to SNL's effort to identify and test coating materials for the prevention, mitigation, and repair of SNF DSCs against potential CISC. The development of an effective corrosion prevention, mitigation, and repair coating for use on SNF storage canisters presents a large technical challenge, but also represents a critically important area of R&D to the back end of the nuclear fuel cycle. The FY20 report [1] provided a detailed description of the specific coating properties designed for application on SNF canisters and provided details into several different coatings and their applicability to coat SNF canisters. In FY 21, SNL has engaged with private industry to create a collaborative R&D program building off the analytical and laboratory capabilities at SNL and the material design and synthesis capabilities of private industry. In doing so, SNL created an MOU that included four companies to date (OPM, WHRD, LUNA, and FC) proposing seven different coating technologies (PEKK; modified polyimide/polyurea with and without phenolic resin modification; modified phenolic resin; silane based polyurethane hybrid with and without a Zn-rich primer; and a quasi-ceramic sol-gel polyurethane hybrid) to be tested, evaluated, and optimized for their potential use for this application. Specific details regarding the coating systems are provided in Section 0. The MOU allows for SNL and the participating industry partner to freely iterate and share technical data for specifications or application process documents when they are developed. The MOU is divided into three phases, which are described in Section 2.1. The work under this MOU will provide an in-depth understanding of the coating systems and their potential use on SNF storage canisters.

A detailed description of the planned experimental activities to be performed by SNL are provided in Section 4. The results of these experimental tests, including physical tests (i.e. adhesion testing), electrochemical (e.g. EIS measurements), and characterization (e.g. microscopy), analyses will be used to identify specific ways to further improve coating technologies toward their application and implementation on SNF canisters. In FY21, SNL has begun baseline testing of the base metal material in accordance with activities under Phase 1 of the MOU. In FY22, SNL will receive coated coupons from each of the participating industry partners and begin characterization, physical, and electrochemical testing following the test plan described in Section 4.4. Following initial testing in FY22, Phases 2 & 3 will commence and will involve experimental evaluations of the coating technologies with respect to their thermal and radiation properties as well as their ability to coat non-pristine surfaces.

The considerations for desired properties under each of the implementation scenarios summarized in this report and FY20 report will provide a roadmap to identify success metrics and areas for further improvement. In addition, the evolution of the coatings tested in this project will be compared with those being explored by PNNL, NEUP, and EPRI.

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