Material Compatibility Study of Coated Metals to Maintain Biocidal Silver in a Spacecraft Potable Water System

Rogelio E. Garcia Fernandez[1]
Jacobs Technology Inc., Houston, Texas, 77058

Stacey Marshall[2]
Aerodyne Industries, Houston, Texas, 77058

and

Niklas Adam[3]
NASA, Lyndon B.Johnson Space Center, Houston, Texas 77058

Ionic silver-based biocide is an option of strong interest for future exploration and has been baselined for several emerging spacecraft potable water systems. A challenge for the use of silver biocide is the loss of ionic silver to wetted materials in the system. Consequently, the design and/or selection of materials that can be used as the wetted system components need specific consideration. As part of the strategy to address this issue, a multitiered approach has been taken to investigate combinations of conventional and non-conventional spacecraft treatment processes and materials. In this study, eleven chemically-resistant polymer coatings were applied as surface treatments onto coupons that were cut out from three types of conventional spacecraft-grade metals (Titanium Grade 2, Inconel 718, and 316L Stainless Steel). The coated metals were immersed in a static 400 parts per billion silver biocide solution at a surface-area-to-volume ratio of 2.0 cm^{-1} and left to soak for predefined periods of time. The concentration of the solution in contact with the coated samples was then analyzed via inductively coupled plasma mass spectrometry, in order to determine the extent to which the coatings provided a barrier to silver loss. This paper summarizes the coating selection process, the configuration of the test, and the performance of each coating-metal combination as a mitigation to silver depletion. The preliminary results demonstrate that select coatings can effectively maintain biocidal silver concentrations. The knowledge acquired through this investigation will be used to assess the feasibility of using these coatings as a robust strategy for maintaining biocidal silver in the water systems. Future studies are planned to assess the specific use of these coatings and to better understand the implications of utilizing this material approach in the future spacecraft potable water system designs.

Nomenclature

Ag+ = silver ion
AgF = silver fluoride
DI = deionized
ICP-MS = inductively coupled plasma mass spectrometry
SA/V = surface area to volume ration
SPWS = spacecraft potable water system

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[1] Life Support Systems Test Engineer, JETS II Contract, Jacobs Engineering, 2224 Bay Area Blvd, Mail Stop: JE01.
[2] Project Manager, JETS II Contract, Crew and Thermal Systems Division, 2101 E NASA Pkwy, Mail Stop: EC3.
[3] Water Technology Engineer, Crew and Thermal Systems Division, 2101 E NASA Pkwy, Mail Stop: EC3.

I. Introduction

Microbial control in wetted components of a Spacecraft Potable Water System (SPWS) continues to be a research topic for further discoussion and development. A practical and efficient multibarrier strategy for microbial control protects the crew, minimizes system maintenance, and reduces the risk of microbial fouling and corrosion. Several groups are investigating ionic silver at concentrations between 200-400 ppb (μg/L) as a common biocide for SPWS aboard different water system architectures (Orion, Lunar Gateway, Lunar Habitats, etc.) due to its effectiveness against a broad spectrum of microorganisms and harmless consumption by humans.[1–6] However, ionic silver losses have been documented in research and development efforts that studied the compatibility between spacecraft-grade materials and silver-based solutions. When silver-containing water is in contact with legacy SPWS metals, there is a propensity for the biocidal form of silver (Ag+) to be depleted from the liquid phase to a surface with Ag+ affinity. These losses can be accentuated in static water conditions and in geometries with large surface area to volume ratios (SA/V), namely small pipe sections, valves, and fittings. Consequently, understanding the fundamental mechanisms that describe Ag+ depletion and the development of mitigating strategies become a matter of priority for the advancement of future SPWS technologies.

The mechanisms that likely describe Ag+ depletion at liquid-solid interfaces are physical and chemical adsorption.[7] During physical adsorption, Ag+ diffuses from the bulk to the amorphous and tortuous features of the adjacent surface due to an established concentration gradient. On the other hand, chemical depletion through electroless deposition consumes the available Ag+ when the corresponding reduction reaction is favorable at a autocatalytic surface (particularly metals) and another constituent in the water serves as a reducing agent (electron provider). Other chemical processes that reduce silver levels occur when Ag+ have the capacity of interacting with superficial functional groups via ion-exchange and complexation mechanisms, but these chemical routes are less expected. Although it is not entirely understood which adsorption pathway is principally responsible for Ag+ depletion during silver biocide experiments, scrutiny of theoretical transport phenomena aspects and empirical results suggest that both physical and chemical adsorption must simultaneously occur to reach the observed depletion rates.

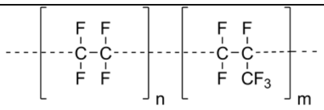
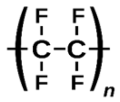
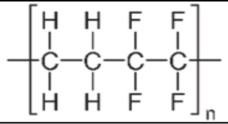
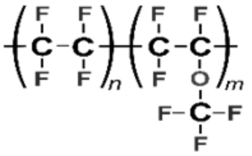
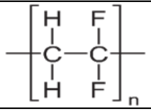
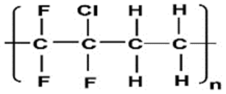
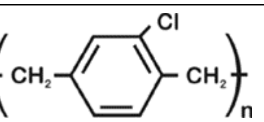
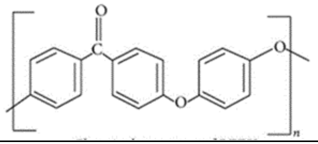
Preceding investigations exposed unconditioned ceramic, plastic, and metallic materials of interest to a 400 ppb silver biocide solution at a 2.0 cm^{-1} SA/V to acquire Ag+ depletion data in stagnant conditions. The preliminary results revealed that the while the silver ion concentration were depleted rapidly, within one-week, upon first exposure to legacy SPWS metals (Titanium Grade 2, Inconel 718, and 316L Stainless Steel), most all of the ceramic and plastic materials maintained target concentrations above 200 ppb over the same period of exposure.[8] Although nonmetallic materials generally exhibit a high degree of compatibilty with silver, these bulk materials may not always be suitable for direct application in SPWS components. An optimized SPWS for silver compatibility would likely require different material-based solutions for different parts of the system that include, but are not limited to, the use of heritage materials, the use of new materials, and/or modified treatments of the materials thereof. Therefore, material treatment and processes must be studied and developed to accommodate material compatibility between wetted components and silver biocide; specially in stagnant conditions during dormancy periods that future vehicles and habitats would need to withstand.

Some of the strategies that could potentially alleviate Ag+ loss by metal surfaces are surface aging, electropolishing, chemical passivation, heat treatment, and coating application.[1,8-11] Among these approaches, surface aging and coating application have been the most studied and have shown material compatibility improvement between silver biocide and metal surfaces in stagnant conditions.[9,10] Surface aging reduces Ag+ depletion by saturating superficial adsorption sites. This apparent saturation is achieved by exposing the metal surface to a highly concentrated silver solution or periodic fills and withdraws of standard silver biocide solutions until an asymptotic limit is observed. Alternatively, coatings offer a dense physical barrier against the penetration of Ag+ and promote a passive state that inhibits any silver reduction reaction. The non-reactive characteristics of coatings may be attributed to the strength of their functional group bond. For instance, most anticorrosion coatings possess single-bond functional groups with the highest bond-dissociation energies; particularly, carbon-fluoride bonds (~513 kJ/mol).[12] Since metals still prevail as the optimal construction material for spacecraft-plumbing components, a coating-metal configuration arises as an attractive design combining the mechanical properties of metals and the passive state of some polymeric materials against Ag+ depletion. Although these techniques have provided promising results, the preliminary studies have not been extensive. Surface aging is a technique that could impose some operational constraints, and only a small number of coatings have been tested. To expand this material-focused approach, the investigation presented in this paper explores a larger set of coatings and tests their capacity to diminish Ag+ depletion on metal surfaces in stagnant water conditions.

II. Materials and Methods

Commercially available coatings were selected from providers that deliver high-performance coatings against corrosion, fouling, high temperatures, chemical weathering and contamination. This selection consisted of products that met the following criteria: 1) extended coating durability, 2) binding compatibility with flight-approved metals, and 3) historical use in applications such as food, potable water, medical products, and spaceflight. Additionally, the capability for candidate coatings to be applied to small diameter tubing was considered, but not required. These specifications led to a list of eleven coatings (see Table 1) for testing: Teflon™ FEP, Teflon™ PTFE, Tefzel®, Teflon™ PFA, Dykor®, Halar®, Dry Film™ RA, Dursan®, SilcoNert 2000®, Parylene C, and PEEK. Each coating from the selection was applied to metal samples of Titanium Grade 2, Inconel 718, and Stainless Steel 316L. This procedure generated a sample matrix consisting of 33 coating-metal combinations for the material compatibility study.

Table 1. List of Coatings for Testing

Branded Name	Chemical Name	Structure
Teflon™ FEP	fluorinated ethylene propylene	
Teflon™ PTFE	Poly-tetrafluoroethylene	
Tefzel®	ethylene tetrafluoroethylene	
Teflon™ PFA	perfluoroalkoxy	
Dykor®	polyvinylidene fluoride	
Halar®	ethylene chlorotrifluoroethylene	
Dry Film™ RA	poly-tetrafluoroethylene	
Dursan®	proprietary	functionalized silica-like structure
SilcoNert 2000®	proprietary	functionalized silica-like structure
Parylene C	chlorinated poly-para-xylylene	
PEEK	polyetheretherketone	

A. Test Methodology

To generate material compatibility data, each coated sample was exposed to silver biocide for a predefined testing period. The silver biocide formulation consisted of silver fluoride (AgF) solution with an initial Ag+ concentration of ~400 ppb. The coated metals were submerged in silver-containing water at SA/V of 2.0 cm^{-1} in accordance with testing conditions from former investigations.[8] This SA/V was selected for comparative purposes and denotes an intermediate value between ratios found in small pipes (~5.0 cm^{-1}) and storage tanks (~0.14 cm^{-1}).[13] Each coating-metal combination incorporated a set of triplicated samples for six different exposure durations: 2 weeks, 4 weeks, 8 weeks, 16 weeks, 32 weeks, and 52 weeks. All groups of samples were prepared using the same AgF stock solution, and their corresponding first-exposure soaking time initiated concurrently.

After a corresponding soaking period ends, water samples are withdrawn from the test article and analyzed for Ag+ concentration. This analysis is carried out through a 7900 Series Agilent Inductively Coupled Plasma-Mass Spectrometer (ICP-MS). The resulting Ag+ levels are compared with the stock solution concentration to determine silver biocide retention data. The intention of the test is to assess if the coatings can keep the Ag+ concentrations between 400 ppb and 200 ppb in the water samples after each soaking period. Percentages close to 100% would indicate that the associated coating successfully impeded Ag+ depletion. Conversely, a coating would deliver poor performance if the water sample maintained less than 50% of the original Ag+ concentration.

Subsequent to the water analysis, the coated samples are re-exposed to a fresh silver biocide solution and the corresponding soaking period is restarted. By carrying out this methodology, more data can be gathered to determine whether the performance of each coating increases or decreases after each silver biocide exposure. The test methdology seeks to conduct up to three re-exposures for all coated samples.

B. Coated Samples

The material compatibility study adopted an elementary geometry for the samples to accommodate adequate coating application, simple sample construction, easy sample inspection, and practical test reproducibility. The uncoated samples were fabricated from Titanium Grade 2, Inconel 718, and Stainless Steel 316L sheets to generate square coupons with 1.35-inches sides and a thickness of 1/16 inch. Additionally, a 3/16-inch circular hole was made at the center of the coupons to insert the fixture that would hold the coupons in the solution during the test. The coupons and their features were machined via computerized water jet to minimize edge-heating while cutting the metal. (By avoiding heat-induced defect on the edge of the coupons, uniformity is ensured for the application of the coatings.) After the coupons were detached from the metal matrix, each coupon was polished with 120-grit sanding media, cleaned with 99% isopropyl alcohol and deionized (DI) water, and air dried for 24 hours.

Once the coupon-manufacturing process was completed, the coupons were shipped to the respective coating applicator. The applicators were instructed to coat all the surfaces of the coupons, with exception of a required edge section on which each coupon needed to be held (a narrow portion on the interior edge of the middle hole, < 1/16 inch) during the application of the coating. More than 600 samples were coated during this process. Furthermore, the coating applicators followed specific packing instructions to minimize damage to the coated coupons during transportation. Immediately after the coated coupons were received, each sample was visually inspected using a 12.5x hand-held magnifier to look for obvious coating damage or poor coating quality. It is important to point out that this material compatibility study did not encompass any meticulous (destructive or non-destructive) mechanical assessment of the coatings before main testing. The visual inspection did not find any alarming defects on the coated surfaces. Therefore, the coating application process was considered successful, and all the coupons were accepted for testing.

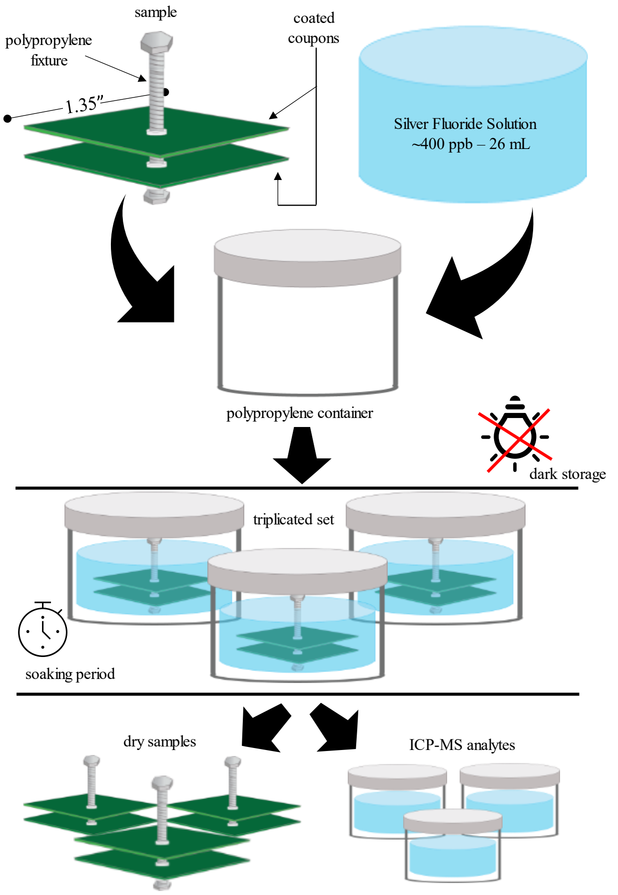


Figure 1. Experimental Setup and Test Overview. *This configuration was utilized for all 33 coating-metal combinations. (The color of the coupons shown in this diagram does not correspond to the actual color of a coating).*

To achieve the selected 2.0 cm^{-1} SA/V for each sample, two coupons were stacked on a non-metal fixture (see Figure 1). The fixture consisted of a polypropylene threaded rod on which the coupons were vertically embedded and separated by washers and nuts of the same material. Since the threaded rod and washers are securely inserted in the middle hole of the coupons, the uncoated holding mark on the edge of the hole was considered to be sealed. The fixture with the two coupons was placed inside a Qorpak® polypropylene container with 26 mL of 400 ppb silver biocide solution. This study selected polypropylene as the material for the fixture and the container since this polymer has shown little to no uptake of ionic silver from water. Before assembling the samples, the coated coupons and the containers were thoroughly rinsed with DI water and allowed to air dry for 24 hours. Each fixture was cleaned using an 18% (volume %) nitric acid solution, flushed with DI water, and allowed to dry for 24 hours.

When all test components and requirements were in place, the test was assembled and initiated in accordance with the test methodology. The containers were filled with special care to reduce any air bubble formation between the coupons. All the samples were stored in the dark to minimize ionic silver precipitation by light-induced photoreduction. Figure 1 displays a general overview of the experimental setup for a generic triplicated set of samples. At each sampling point, the containers are removed from the dark confinement, and the coupon stacks are transferred to an ambient temperature storage location for air drying while the analytes are transported to the water analysis site in the original container. Each container is wrapped with aluminum foil to prevent photo-reduction of dissolved silver while the samples waited for analysis.

Moreover, the material compatibility study also incorporated reference samples. However, these control samples only comprised of silver biocide solution and the non-metal fixture without coupons. Based on the well-documented depletion of silver by unconditioned metal coupons, uncoated samples were not attempted. Preliminary testing with bare coupon has shown that the machined coupons can completely deplete silver from a 400-ppb solution within a one-week period in stagnant conditions. Consequently, by omitting uncoated samples, fewer materials were needed, and less analysis time was required. The control samples provided information describing how the concentration of the silver solution varied over time when the solution only interacted with the container and the fixture material.

III. Material Compatibility Results

As of the acceptance date of this conference paper, the first portion of the material compatibility study has proceeded past the 32-week sampling point. Although some sample groups have been subjected to silver-containing water more than once, this paper only compiles the results from water analytes collected after the first silver biocide exposure of the coated coupons. This section compiles the silver-retention results based on ICP-MS data for each coating-metal combination. The time-series data was interpreted to visualize how much silver was retained in solution due to the associated coating after each soaking stage. The upkeep of Ag+ levels was reported as an averaged percentage of the initial concentration for each triplicated analyte. For instance, a 50% would indicate that half of the Ag+ were depleted, and the other half remained in solution. Bar graphs are employed to show the averaged metrics and include error bars that only represent the standard deviation of the triplicated analytes and do not incorporate any quantification of systematic or random error. Figures 2-12 display the material compatibility results for each coating in the same order as shown in Table 1. Some results exhibited values that were higher than 100% since the analytical equipment provided readings that were higher than the initial concentration of the stock solution. This anomaly may be attributed to the inherent variability of the available instrument which is considered to be at least +/- 10%.

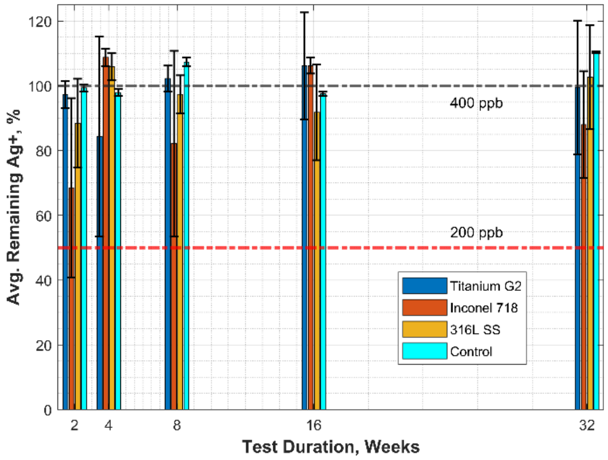


Figure 2. Remaining Ag+ concentration from water samples in contact with Teflon FEP™-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

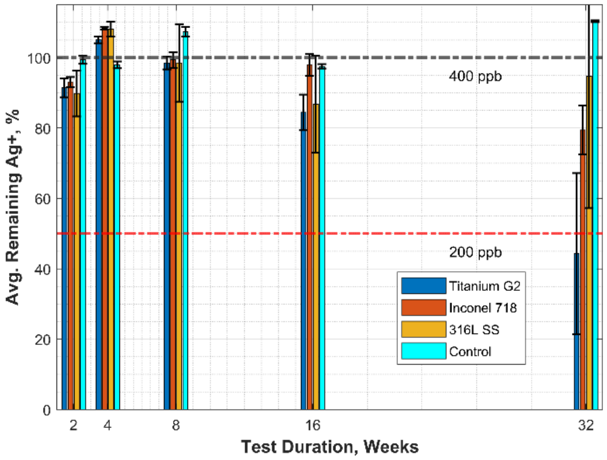


Figure 3. Remaining Ag+ concentration from water samples in contact with Teflon PTFE™-coated coupons after 1st Silver Biocide exposure, 2.0 cm^{-1} SA/V.

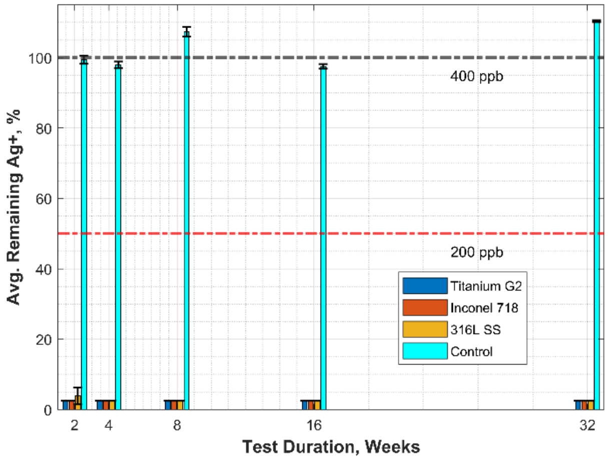


Figure 2. Remaining Ag+ concentration from water samples in contact with Tefzel®-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

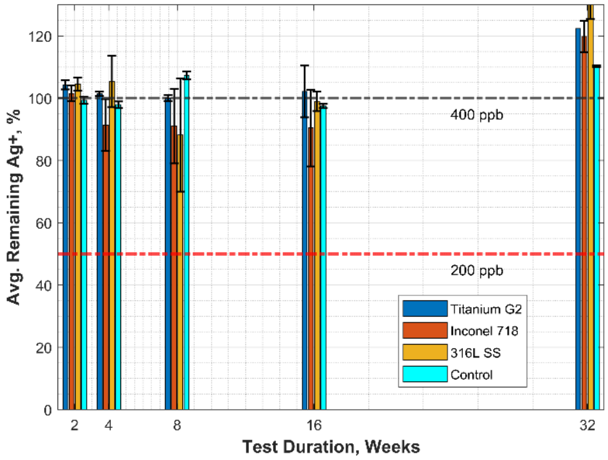


Figure 3. Remaining Ag+ concentration from water samples in contact with Teflon™ PFA-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

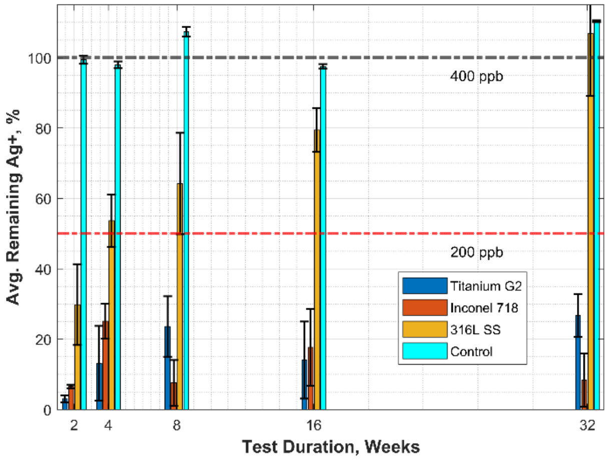


Figure 4. Remaining Ag+ concentration from water samples in contact with Dykor®-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

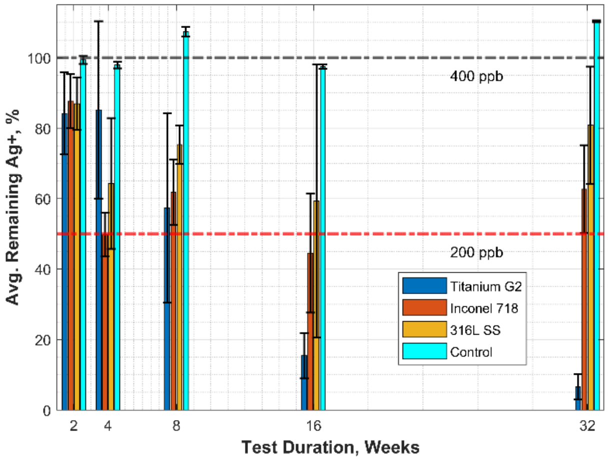


Figure 5. Remaining Ag+ concentration from water samples in contact with Halar®-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

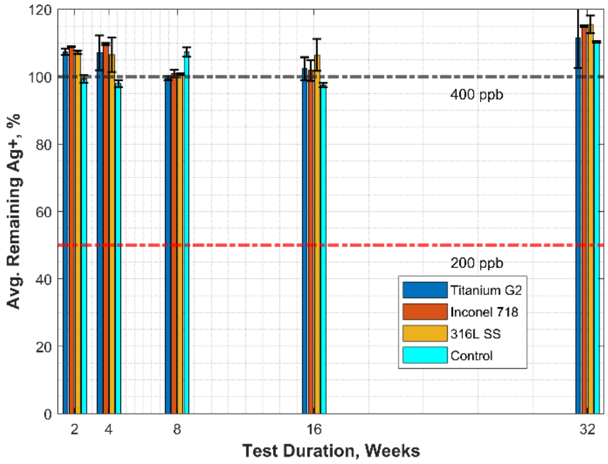


Figure 6. Remaining Ag+ concentration from water samples in contact with Dry Film™ RA-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

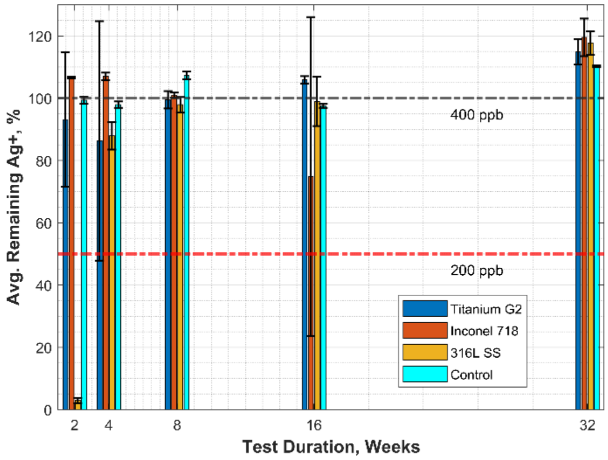


Figure 7. Remaining Ag+ concentration from water samples in contact with Dursan®-coated coupons after 1st silver biocide exposure, 2.0 cm^{-1} SA/V.

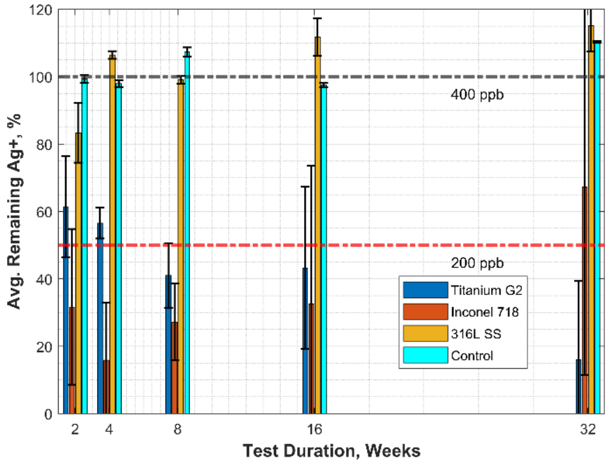


Figure 8. Remaining Ag+ concentration from water samples in contact with SilcoNert 2000®-coated coupons after 1st silver biocide exposure, 2.0 cm-1 SA/V.

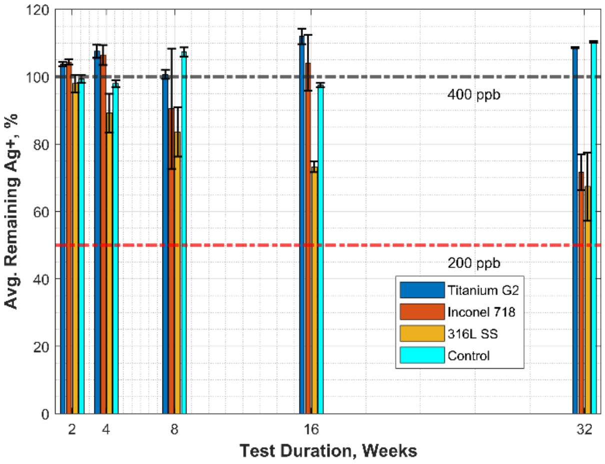


Figure 9. Remaining Ag+ concentration from water samples in contact with Parylene C-coated coupons after 1st silver biocide exposure, 2.0 cm-1 SA/V.

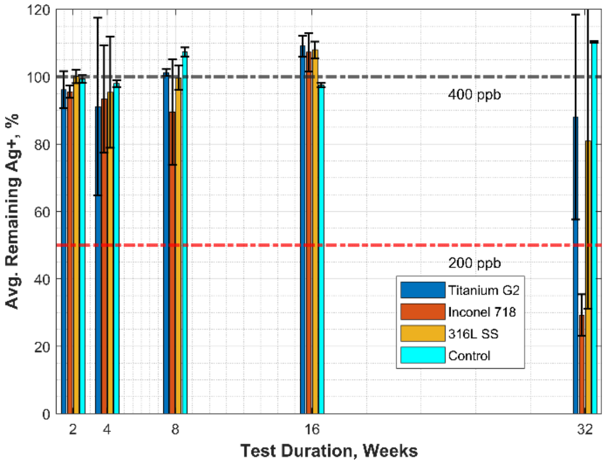
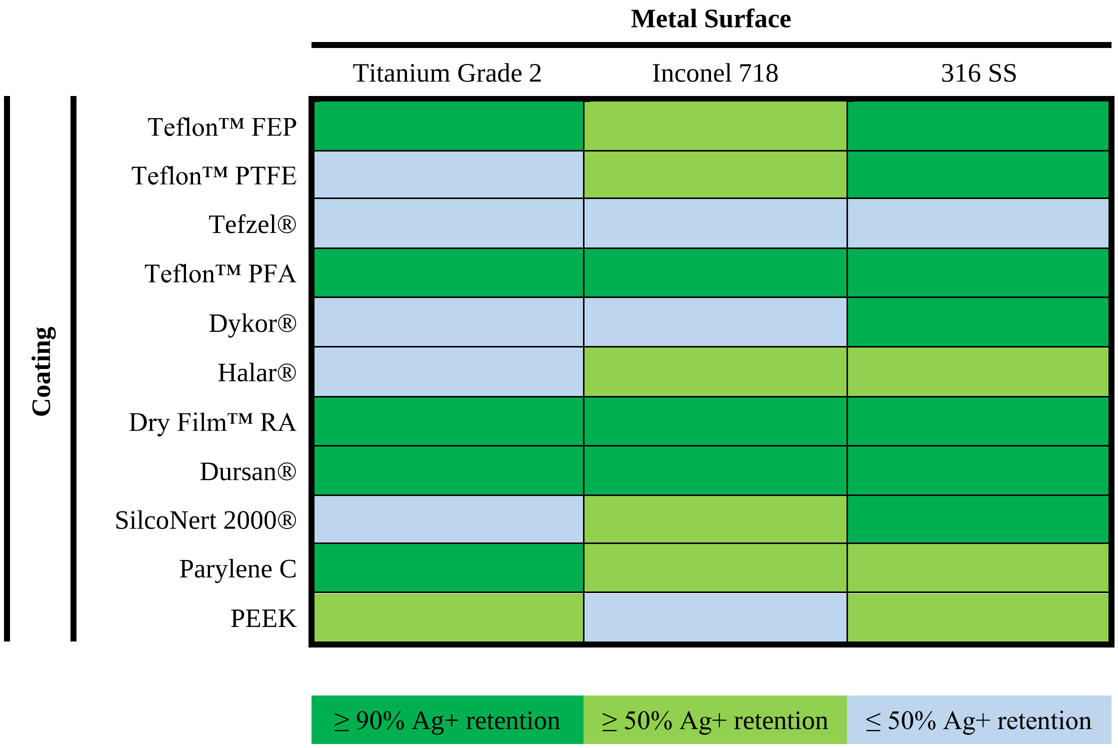


Figure 10. Remaining Ag+ concentration from water samples in contact with PEEK-coated coupons after 1st silver biocide exposure, 2.0 cm-1 SA/V.

In general, Figures 2-12 show that 24 out of the 33 coating-metal combinations allowed Ag+ levels to be maintained above the 200 ppb limit after 32 weeks. Each type of coating performed relatively the same on all the metals, with a few exceptions. This trend may indicate that the performance of these coatings do not heavily dependent on the type of metal. Nevertheless, the coatings demonstrated better performance on 316L Stainless Steel compared to their performance on the other two metals in all data points, with a success rate of 17% for maintaining higher Ag+ levels. Only two coatings showed significant improved performance on specific metals: Dykor® and SilcoNert 2000® reduced more Ag+ depletion on 316 L Stainless Steel than on Titanium Grade 2 and Inconel 718 surfaces. Furthermore, PEEK, Dry Film™ RA, Teflon™ FEP, Teflon™ PFA, and Parylene C were (in decreasing order) the top-performing coatings on all metals, keeping Ag+ levels above 90% up to 16 weeks. After 32 weeks, the performance of PEEK and Parylene C decreased below 90%. Dry Film™ RA maintained the best performance consistency at each sampling point and provided the most precise metrics. Figures 7 is the only chart that reveals a consistent concentration decrease over time up to 16 weeks. However, Halar® improved performance at the 32-week mark for Inconel 718 and 316L Stainless Steel. The coating that allowed the most Ag+ loss was Tefzel®; all water analytes from the Tefzel® samples generated ICP-MS readings below the detection limit (≤10 ppb). This result makes Tefzel® the worst-performing coating from this first exposure experiment. Nevertheless, preliminary results from silver biocide sequential-exposure (not included in this paper) tests started showing that this coating can signifiantly improve performance (above 50% retention) after an initial soaking period with silver-containing water. Forthcoming pulbications will prove results from the study of coating performance during sequential silver biocide exposures. Table 2 summarizes the results of the Ag+ retention analysis from the 32-week samples. After 32 weeks, Dry Film™ RA, Teflon™ PFA, Dursan®, Teflon™ FEP, and Parylene C retainined the most Ag+ among all coatings.

Table 2. Summarized 32-week Ag+ Retention Results



	Metal Surface		
Coating	Titanium Grade 2	Inconel 718	316 SS
Teflon™ FEP	🟩	▩	🟩
Teflon™ PTFE	🟦	▩	🟩
Tefzel®	🟦	🟦	🟦
Teflon™ PFA	🟩	🟩	🟩
Dykor®	🟦	🟦	🟩
Halar®	🟦	▩	▩
Dry Film™ RA	🟩	🟩	🟩
Dursan®	🟩	🟩	🟩
SilcoNert 2000®	🟦	▩	🟩
Parylene C	🟩	▩	▩
PEEK	▩	🟦	▩

🟩	▩	🟦
≥ 90% Ag+ retention	≥ 50% Ag+ retention	≤ 50% Ag+ retention

While analyzing these results, the underlying assumption prescribed that the coated surfaces were primarily attributed to silver loss. The metrics from the control samples support this hypothesis since the Ag+ levels did not dramatically decrease in the absence of the coated coupons. Although some results included a significant standard deviation, the authors find the metrics compelling and sufficient. The broad error bars, that here present the sample standard deviation, were anticipated since the triplicated samples per coating-metal combination constituted a statistically small dataset. Moreover, some charts exhibited results that exceeded control metrics and the limit associated with the initical Ag+ concentration. The rationale that explains this outcome is mainly attributed to instrument drift during ICP-MS measurements. Release of foreign Ag+ by the pristine coating or metal surface, although plausible, was not likely to occur at the observed levels. Therefore, the coated sample (and the container & coupon fixture) could not be responsible for Ag+ levels exceeding control metrics.

It is important to note that no perceptible coating damage or delamination was observed after each coating completed a soaking period. Analysis via Scanning Electron Microscope with Energy Dispersive Spectroscopy did not detect any appreciable change in the chemistry of the coatings or obvious silver localities on the coating. Future publications might elaborate more on coating morphology analyses.

IV. Conclusion and Forward Work

The Lyndon B. Johnson Space Center Water Technology Development Group has initiated a broad material compatibility study for the advancement of silver biocide compatibility with SPWS materials of construction. This research and development effort has generated essential knowledge to better assess coatings as a mitigating strategy for Ag+ loss by metal surfaces. The preliminary results demonstrated that a significant number of coating-metal combinations maintained silver biocide concentrations above 200 ppb after 32 weeks of testing. Previous testing showed that uncoated metal coupons completely deplete Ag+ levels in a one-week period; however, the results from this study indicate that some coatings are not only capable of completely inhibiting Ag+ loss but many also significantly improve the silver loss performance over the untreated base metal. As the tests proceed, more data will allow for better evaluation of this material strategy (specially, after the 52-week data is collected for all sequential exposures). So far, this investigation revealed that Dry Film™ RA, Teflon™ PFA, Dursan®, Teflon™ FEP, and Parylene C substantially reduced Ag+ depletion by surfaces comprising of Titanium Grade 2, Inconel 718, and 316L Stainless Steel. Nevertheless, this recommendation should be reevaluated after these materials complete all testing stages. Future ICES submissions will provide updates regarding this material compatibility study.

After the coated samples conclude all soaking periods and sequential silver biocide exposures, this investigation seeks to down select and apply the best-performing coatings on curved surfaces associated with high SA/V magnitudes to further study coating performance. It is recognized that not all coatings from this selection can be efficiently applied to the water system surfaces associated with high SA/V. From this coating selection, the only coatings that can be effectively applied to high-SA/V geometries are Dry Film™ RA, Dursan®, SilcoNert 2000®, Parylene C, and PEEK (intermediate & low SA/V). The main limition for the application of the other coatings on this list is direct line of sight during conventional spraying, as supposed to high surface curvature. Therefore, in some cases additional engineering may be required for implementation of a coating in certain applications. Another future objective that this material compatibility study attempts to achieve is the meticulous analysis of organic carbon release by the coatings. To adequately quantify whether the coatings leach a significant amount of organic carbon into the silver-containing water, the experimental setup must be modified to minimize any carbon contamination by non-test articles.

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