Quantitative Nondestructive 5XXX Aluminum Material Assessments to Reduce Total Ownership Cost

Overview

The U.S. Navy is using 5XXX aluminum as a critical construction material in the design and construction of state-of-the-art Navy ships. The current U.S. Navy shipbuilding plan will grow the size of the fleet with significant aluminum structural components to approximately 170 vessels by 2040, including: 32 Littoral Combat Ships (LCS), 20 Future Frigates (FF), 22 Guided Missile Cruisers (CG), 12 Aircraft Carriers (CVN), 11 Joint High Speed Vessels (JHSV), and 73 Ship to Shore Connectors (SSC) to replace the 91 Landing Craft Air Cushion (LCAC) vessels. Ships and vessels constructed from 5XXX series aluminum are susceptible to a metallurgical phenomenon called sensitization. Sensitization is the formation of magnesium rich beta-phase precipitates at material grain boundaries as the result of exposure to elevated temperatures for extended periods of time. These beta-phase precipitates are anodic to the surrounding aluminum matrix, and when exposed to a corrosive environment sensitized material will experience intergranular corrosion (IGC). When external tensile stress is applied to material that has experienced IGC, stress corrosion cracking can result. Welding to sensitized material in support of repair, maintenance, and modernization activities requires specific critical welding procedures and in some cases the application of cold working technologies or wholesale material replacement depending on the degree of sensitization (DoS).

Quantitative nondestructive 5XXX aluminum material assessments will significantly reduce costs associated with two specific aspects of maintenance, modernization, and repair activities involving sensitized aluminum, resulting in significant total ownership cost savings across the aluminum fleet:

1. Material assessments prior to repair, maintenance, and modernization availabilities will facilitate more accurate work specifications, which will reduce expensive growth work and yield more predictable availabilities.
2. Quantitative nondestructive 5XXX aluminum material assessments can be performed in lieu of time consuming and expensive destructive sensitization testing (ASTM-G67).

The importance of implementing innovative approaches to reduce total ownership costs associated with the repair, maintenance, and modernization of aluminum vessels is best exemplified by the problems and significant increases in total ownership costs associated with the repair of a significant number of sensitization and fatigue related superstructure cracks across the 22-ship Ticonderoga class guided missile cruisers. The high cost of aluminum crack repair and significant number of CG superstructure cracks in addition to the difficulties associated with working on sensitized aluminum has added several hundred million to the total ownership costs of the CG class ships.

ABSTRACT

With the expected service life of U.S. Navy ships extending to 30-50 years and beyond, the use of innovative technologies that enable predictable execution of maintenance, modernization, and repair activities is critical to reducing the total ownership costs associated with the extended service life of these ships. If in the upcoming years the entire aluminum fleet combined, including Littoral Combat Ships (LCS), Future Frigates (FF), Aircraft Carriers (CVN), Joint High Speed Vessels (JHSV), Landing Craft Air Cushion (LCAC), and Ship to Shore Connectors (SSC), experience the same amount of destructive testing costs, sensitization-related cracking, and growth work per year as the CG class currently experiences, use of quantitative nondestructive aluminum sensitization material assessments could potentially reduce fleet-wide total ownership costs by up to $30 M per year [Dunn, 2015a].
Quantitative Nondestructive 5XXX Aluminum Material Assessments to Reduce Total Ownership Cost

With an additional 20 years of service life remaining for many of the CG class ships, it is conservative to estimate that stress corrosion cracking and the difficulties associated with performing work on sensitized aluminum will continue to significantly increase total ownership costs. In addition to the increase in total ownership costs associated with stress corrosion crack repair, data from NAVSEA PMS 407 CG modernization installations show that aluminum structural ship alterations experience between 20% to 60% growth during execution [TIPS, 2012]. It is estimated that approximately half of this growth work is associated with plans and specifications that do not correctly scope the work associated with sensitized aluminum [TIPS, 2012]. It is anticipated that use of the DoS Probe to support the planning process will improve the accuracy of the resulting work specifications, and reduce growth work and help yield more predictable availabilities.

Use of quantitative nondestructive material assessments on CG class ships can potentially save up to $2M per year in destructive ASTM G67 testing costs (300 tests per year baseline) and up to an additional $15M per year in reduced growth work by performing material assessments prior to aluminum ship alteration and modernization activities [Dunn, 2015a]. If in the upcoming years the entire aluminum fleet, including LCS, FF, CVN, JHSV, and LCAC/SSC vessels combined, experience the same amount of destructive testing costs, sensitization related cracking, and sensitization related ship alteration growth work per year as the CG class, use of quantitative nondestructive aluminum sensitization material assessments could potentially reduce fleet-wide total ownership costs by up to $30M per year [Dunn, 2015a].

The Degree of Sensitization (DoS) Probe (Figure 1) is a technical readiness level (TRL) 9 nondestructive tool currently approved by NAVSEA 05 for quantitative material assessment of the 5456 series aluminum alloys found on CGs, Dock Landing Ships (LSDs), and LCACs. To date the DoS Probe has been used to conduct over 1,400 measurements on various CGs in support of modernization, maintenance, and repair efforts. PEO LCS and the Office of Naval Research (ONR) are currently evaluating a research effort to calibrate the DoS Probe for use on the remaining aluminum alloys used on the LCS-1 and LCS-2 variants, FF (Future Frigate), JHSV, and SSC class vessels.

NONDESTRUCTIVE MATERIAL ASSESSMENTS TO REDUCE TOTAL OWNERSHIP COST

There are four specific use cases where quantitative nondestructive material assessments can significantly reduce total ownership costs through the development of more accurate work specifications that result in predictable execution of maintenance, modernization, and repair availabilities:

1. Major Modernization Planning
2. Third Party Work Specification Development
3. Real Time Support of Ship Repairs
4. Support of Ship Maintenance Activities

MAJOR MODERNIZATION PLANNING

The DoS Probe can be used during the planning phase to significantly reduce growth work experienced during execution by providing the repair facility with a more accurate work package and avoiding costly and slow use of conventional destructive aluminum testing during execution. For example, in Figure 2 the diagonally hashed areas indicate the originally planned aluminum insert. The DoS Probe was used to quantify the degree of sensitization in each area adjacent to where an insert was planned. In areas that tested below the sensitization threshold (green circle) the original work plan was adequate. In areas where inserts were planned adjacent to moderately sensitized material (yellow circle) the work plan was modified to include the use of Ultrasonic Impact Treatment (orange hashed line). In one area, the entire plate where they were planning to perform the insert was found to be highly sensitized (red circle).

In this case, the work plan was updated to include a larger insert (pink shading). More accurate work planning will avoid costly and time-consuming execution growth work associated with crack chasing and destructive testing.

To support PMS 407 planning work for major CG modernization events between June 2013 and September 2015, DoS Probes were used to take approximately 1,400 measurements...
Quantitative Nondestructive 5XXX Aluminum Material Assessments to Reduce Total Ownership Cost

Third party work specification development

Third party advance planning of repair efforts can also benefit from the use of DoS Probes in a similar way that major modernization planning efforts benefit from the use of the technology to produce more accurate work plans resulting in less growth work and more predictable execution availabilities.

Real time support of ship repairs

Nondestructive DoS Probe material assessments can also reduce repair times and costs when used in lieu of destructive testing in support of ongoing repair activities as urgent testing needs arise. The two-day turnaround associated with ASTM G67 test facilities located at Regional Maintenance Center laboratories and up to 10-day turnaround associated with ASTM G67 testing at the Naval Surface Warfare Center Carderock Division (NSWCCD) laboratories can result in delay and disruption when tests are performed as part of an ongoing ship repair effort [TIPS, 2012].

Support of ship maintenance activities

Ships and regional maintenance centers can benefit from the cost and time savings associated with DoS Probe technology relative to ASTM G67 destructive testing [TIPS, 2012]. The nondestructive nature of DoS Probe testing is more suitable for conducting measurements at the RMC facility prior to ship deployments and scheduled work availabilities.

Technical background

To maximize the stability and, in some cases, speed of naval vessels, aluminum-magnesium (5XXX) alloys are often used to construct ship superstructures and more recently the hull and superstructure of the Independence variant of the Littoral Combat Ships. The primary metal, aluminum, offers the advantage of being lightweight; adding magnesium to the aluminum creates an alloy that is both lightweight and strong. To understand how sensitization, intergranular corrosion (IGC), and stress corrosion cracking (SCC) impact the U.S. naval vessels constructed from 5XXX aluminum, the composition and basic material properties of 5XXX aluminum alloys need to be explained.

5XXX aluminum material properties

A majority of 5XXX aluminum alloys used in ship construction are produced via a rolling process. The rolling process (Figure 3) can result in variations in material grain size and microstructure.

The current destructive sensitization testing standard (ASTM G67) is known to be sensitive to variations in sample grain size and microstructure, so development of a comparable nondestructive testing mechanism required these sensitivities to be fully understood. Analysis via Barkers etch of approximately 50 samples of CG fleet material revealed that a large range of grain sizes and microstructures can be found in the fleet. Material grain sizes observed (Figure 4) ranged from small grains less than 25 μm in size to large grains over 200 μm in size [Kelly, 2011].

All three microstructure classifications; recrystallized, partially recrystallized, and unrecrystallized, were also observed in the same analysis of CG fleet material (Figure 5). Recrystallized samples have round compact grains on the rolled (LT) surfaces as well as throughout the thickness of the material. Partially recrystallized samples have longer “pancake” shaped grains through the thickness of the material. Unrecrystallized samples have longer “pancake” shaped grains on the rolled surfaces (LT) as well as throughout the thickness of the material. It should be noted that smaller gauge material is more subject to recrystallization and partial recrystallization as a result of the rolling process. Large gauge (>0.5”) material is typically either unrecrystallized, or in some cases can be partially recrystallized.

5XXX aluminum alloy composition

The primary alloying element in 5XXX aluminum (Al) alloys is magnesium (Mg). During production, highly controlled heat treatments are used to evenly distribute magnesium (Mg) in the aluminum (Al) matrix. Different alloys in the 5XXX
series contain varying amounts of Mg ranging from ~3.5% in 5086 to ~4% in 5083 up to ~5% in 5456.

**FIGURE 5.** 5XXX Microstructure variations. NSWCCD Micrographs.

**5XXX ALUMINUM ALLOY SENSITIZATION**

The evenly distributed state of the Mg within the Al matrix is thermodynamically metastable and exposure to even mildy elevated temperatures for extended periods of time will cause the magnesium to form beta-phase \((\text{Mg}_2\text{Al}_3)\) precipitates. The formation of these beta-phase precipitates along the grain boundaries as a connected network is called sensitization. Figure 6 shows a side-by-side comparison of unsensitized 5456 Al (left) vs. sensitized 5456 Al (right). As shown in the figure, the sensitized sample contains a connected network of beta phase precipitates along the grain boundaries that were visibly etched when exposed to a phosphoric acid etch.

At a high level, the rate of sensitization is primarily a function of four factors: thermal exposure, alloy composition (% Mg), material temper, and the material grain size and microstructure. Assuming equivalent thermal exposures, tempers, grain sizes, and microstructures 5XXX Al alloys containing higher amounts of magnesium will sensitize faster than 5XXX Al alloys with lesser amounts of magnesium. For example, 5456 (~5% Mg) will sensitize faster than an equivalent 5083 (~4.0% Mg) sample, and 5083 will sensitize faster than an equivalent 5086 (~3.5% Mg) sample when exposed to the same thermal conditions (Figure 7).

**FIGURE 6.** Left: Unsensitized Al 5456, Right: Sensitized Al 5456. NSWCCD Micrographs.

**FIGURE 7.** 5XXX Al sensitization rates at 100°C for recrystallized material. [Davis, 2010].

The Naval Surface Warfare Center Carderock Division (NSWCCD) has an ongoing effort to predict sensitization rates of various 5XXX aluminum alloys in the fleet. As a part of this effort, two sample racks holding six samples each were mounted (one vertical, one horizontal) on a deployed Navy vessel. Test samples include both 5456 and 5083 alloys with various coating systems applied. The temperature of each test specimen has been measured and recorded by an attached thermocouple every 20 minutes. The collected temperature data was then fed into a predictive model developed by NSWCCD to predict sensitization rates for different 5XXX alloys based on the recorded thermal exposures. Based on this data it is estimated that recrystallized 5083 will reach sensitization levels above 25 mg/cm² after approximately 7 to 10 years of fleet exposure [Golumbfskie, 2010] and 5456 will sensitize after approximately 4 years of fleet exposure [Golumbfskie, 2009].

**INTERGRANULAR CORROSION OF SENSITIZED 5XXX ALUMINUM ALLOYS:**

The beta-phase \((\text{Mg}_2\text{Al}_3)\) precipitates contain approximately 38% Mg which is significantly higher than the Al matrix, which for Al 5456 contains only approximately 5% Mg. The compositional relationship between beta-phase and matrix Al is shown in Figure 8.

**FIGURE 8.** Beta-phase = 38% Mg vs. Al Matrix = 5% Mg (5456). [Dunn, 2014].

Elemental Mg is thermodynamically less stable and kinetically more active than elemental Al. These characteristics make Mg more susceptible to dissolution in low and neutral pH environments. The beta-phase \((\text{Mg}_2\text{Al}_3)\) behaves more like...
Mg than Al and will dissolve rapidly in seawater environments. This difference in dissolution behavior, combined with the fact that beta-phase preferentially forms on grain boundaries during service, leads to the preferential corrosion of those grain boundaries, which is termed intergranular corrosion (IGC).

**Stress Corrosion Cracking (SCC)**

Stress corrosion cracking will occur if a specific set of material properties and environmental conditions are present. As illustrated in Figure 9, sensitized material is one of the conditions that contributes to SCC of aluminum alloys. The sensitized material then needs to be exposed to a corrosive environment and IGC needs to initiate corrosion along grain boundaries. Lastly, a tensile stress needs to be applied to the IGC affected material to form a stress corrosion crack.

It should be noted that material sensitization alone does not result in stress corrosion cracking. For example, there have been instances on CG flight decks where material has tested as sensitized but the lack of significant tensile stresses has historically not resulted in stress corrosion cracking problems in this area.

Figure 10 provides a conceptual summary of how sensitization relates to intergranular corrosion and stress corrosion cracking.

**Destructive Sensitization Testing (ASTM G67)**

Until recently, destructive Nitric Acid Mass Loss Testing (NAMLT) via the ASTM G67 test standard has been the Navy’s only option to quantitatively assess the degree of sensitization of 5XXX Al alloys. ASTM G67 involves excising a coupon from the ship’s structure, immersion of a portion of this coupon in 70% nitric acid for 24 hours at a tightly controlled temperature, and measuring the mass loss per unit area (mg/cm²) resulting from intergranular corrosion. The problems with the G67 test are that it is destructive, requires laboratory testing, is expensive, and is labor and time intensive.

The average direct cost associated with each ASTM G67 test location can be as high as $6,800 per test [TIPS, 2012]. Total testing cost consists of cutting the sample hole, machining the sample, laboratory testing costs, and in some cases inserting weld repairs to fix the hole, a required fire watch, the removal and replacement of interferences, and repainting. Small insert repairs (<12") requiring aluminum welding average $5,000 per repair and can increase dramatically if significant interferences need to be removed and replaced [TIPS, 2012]. Laboratory ASTM G67 testing fees average $200 and additional costs associated with sample coupon harvesting and sample machining can increase costs to between $1,000 and $1,800 per sample [TIPS, 2012]. This cost estimate does not include indirect costs, readiness impacts, or delay and disruptions associated with the two-day turnaround associated with ASTM G67 test facilities located at Regional Maintenance Center laboratories, and up to a ten-day turnaround associated with ASTM G67 testing at
the Naval Surface Warfare Center Carderock Division laboratories. The worst case scenario is when sensitized material is detected and an additional destructive sample needs to be taken, incurring another two to ten days of delay and disruption to scheduled work packages, and this process repeats until material capable of being welded is detected.

**ASTM G67 LABORATORY PROCEDURE SUMMARY**

From the 4” diameter circular coupon excised from the ship, two specimens 2.00” by 0.25” by sample thickness (up to 1.00”) are machined. These samples need to be machined such that the 2” dimension is along the “L” (rolling) direction of the material. Determining the rolling direction can be accomplished via performing either Barker’s or phosphoric acid etching (if sensitized) prior to machining the samples. Machining the samples in alignment with the rolling direction helps standardize the surface area to grain profile ratio among all ASTM G67 specimens.

**FIGURE 13.** ASTM G67 Machined Specimens.

Once the two samples have been machined and cleaned, the surface area of each sample is calculated by measuring each dimension to the nearest 0.001”. Each sample is then weighed to the nearest 0.1 mg to determine an initial mass value. Each sample is then carefully placed in vials containing 70% reagent-grade nitric acid in such a way that none of the major surfaces are in contact with the sides of the container. The vials are placed in either an oven or a water bath with a tightly controlled temperature of 30°C ± 0.1°C for exactly 24 hours. When each sample is removed from the nitric acid solution, it is rinsed with water while the test operator brushes the sample with a stiff plastic brush (an ultrasonic cleaning bath can also be used for more uniform removal of particulates). The post NAMLT mass of each sample is then recorded to the nearest 0.1 mg and the mass loss in mg/cm² is calculated using the initial surface area and the mass loss that occurred as a result of the NAMLT test.

**ASTM B928 RESULTS AND REPAIR GUIDANCE**

ASTM B928 provides guidelines for ASTM G67 results when purchasing material for ship construction. Section 9.3 of ASTM B928 outlines the criteria for pass, fail, and questionable intergranular corrosion resistance according to ASTM G67 testing. Based on the ASTM B928 standard, 5XXX Al alloys that exhibit less than or equal to 15 mg/cm² pass the test. Samples that test between 15 mg/cm² and 25 mg/cm² shall be deemed questionable and subjected to additional metallographic testing, and samples that exhibit greater than or equal to 25 mg/cm² of mass loss shall be rejected (Figure 16).

**FIGURE 15.** NSWCCD ASTM G67 Test Setup. [Golumbfskie, 2012]

The Navy has created their own guidance on the weldability of sensitized 5XXX aluminum based on ASTM G67 testing [NAVSEA, 2013]. Under the Navy’s guidance, material that tests between 0 and 20 mg/cm² can be welded using normal aluminum welding standards; material that tests over 20 mg/cm² must meet critical welding requirements. Material that tests between 30 and 60 mg/cm² must have the weld treated with cold working treatments to prevent re-cracking, and
material that tests over 60 mg/cm² must be replaced. It should be noted that even material with a DoS of 25 mg/cm² is considered sensitized according to the ASTM B928 standard.

**ASTM G67 MICROSTRUCTURE BIAS**

Exposure to 70% nitric acid at 30°C creates an extremely corrosive environment in which anodic beta-phase precipitates corrode preferentially to the surrounding Al matrix.

The total mass loss resulting from ASTM G67 testing is the sum of the mass loss associated with the corrosion of the beta-phase precipitates plus the mass loss resulting from associated grain fall out.

The strong nitric acid solution corrodes away exposed beta-phase precipitates on all six sample surfaces, resulting in some initial mass loss attributed solely to the dissolution of the beta-phase precipitates. To the extent these beta-phase precipitates exist in a connected network at grain boundaries, once enough intergranular corrosion (IGC) has taken place the entire Al matrix grain will fall out, significantly increasing total mass loss.

Recrystallized grain structures require only a small amount of grain boundary beta-phase precipitates to initiate Al matrix grain fall out, whereas unrecrystallized grain structures require a much more significant network of connected grain boundary beta-phase precipitates for IGC to result in grain fall out. In addition, it is easier for the more equiaxed recrystallized grains to be removed from the surface, as the “pancake” shaped grains can remain interlocked even after most of the beta-phase has been dissolved. For these reasons, recrystallized grain structures will yield higher mass loss values with significantly lower concentrations of beta phase on the grain boundaries than unrecrystallized grain structures (Figure 17).

This bias can be observed visually by cross sectioning two ASTM G67 samples of similar mass loss value with different microstructures (Figure 18). The machined edge of the recrystallized sample is no longer visible, meaning that wholesale material loss contributed to the mass loss value. Sample dimensions can be measured post exposure and volumetric decrease can be correlated with microstructure.

The same bias with respect to microstructure can be observed when applying sensitization heat treatments to samples with recrystallized and unrecrystallized microstructures and observing the time it takes for the samples to reach the same ASTM G67 mass loss values (Figure 19).

The ASTM G67 bias with respect to microstructure is understood by the NAVSEA 05 technical community and was taken into consideration when the SXXX Al repair guidelines (Figure 16) were created. Based on empirical results from field tests, it appears that the most severe SCC onboard CGs happen when recrystallized material becomes sensitized.

**ASTM G67 STANDARD ERROR BARS**

The ASTM G67 standard defines error bands for the ASTM G67 test procedure at three specific points: 8 ± 3.9, 30 ± 5.5, 44 ± 6.6 (all in mg/cm²). Using these points, a best fit line can be extrapolated with the following equation: \(y = 13.363x - 43.939\), which when solved for \(y\) will give the + error band value for any \(x\) value (Figure 20). For example, a reported ASTM G67 test value of 60 mg/cm² can really be anywhere between 53 mg/cm² and 67 mg/cm². Such large error bands can make repair and maintenance decisions based on the repair guidance threshold values shown in Figure 16 difficult.

ASTM G67 contains additional potential sources for intra and inter laboratory variability that are not captured in the standard error bars. Testing and machining the ASTM G67 samples along the L direction of the material is a procedural step...
Quantitative Nondestructive 5XXX Aluminum Material Assessments to Reduce Total Ownership Cost

that was only followed during testing for CGs when it could be easily determined. L direction testing is complex and requires etching the sample and examining the sample prior to machining it. Limited test data show that not machining ASTM G67 test samples along the L direction can increase error associated with ASTM G67 testing by an additional 10% [Bovard, 2011]. Error associated with lack of specified L direction machining is likely more significant in unrecrystallized material because the long “pancake” grains align with the L direction of the material. Another potential source of variation in ASTM G67 testing is during the cleaning of the samples after the exposure to nitric acid. The standard calls for cleaning with a stiff plastic brush. This introduces the potential for operator variation where some test operators may not scrub the sample vigorously enough to remove all lose particles and could result in lower mass losses. One of the most significant sources of potential variation is found in the calibration and ability of the oven or water bath to tightly control the temperature of the samples while they are exposed to nitric acid. The standard calls for cleaning with a stiff plastic brush. This introduces the potential for operator variation where some test operators may not scrub the sample vigorously enough to remove all lose particles and could result in lower mass losses. One of the most significant sources of potential variation is found in the calibration and ability of the oven or water bath to tightly control the temperature of the samples while they are exposed to nitric acid to exactly 30°C. Even a 1°C difference in temperature from lab to lab, or drift in temperature during a measurement in the same lab, can result in drastically different mass loss results as temperature has an exponential effect on the rate of the ICG corrosion reaction taking place.

To further study potential inter-laboratory variation, ALCOA organized a Round Robin with nine different ASTM G67 labs [F. Bovard and J. Moran, 2010]. The initial results in Figure 21 show that when you add in variability among different testing laboratories, the error band for a 45 mg/cm² sample increases from ±6.7 mg/cm² (±15%) to ±11 mg/cm² (±24%). In the worst case, results ranged from one lab reporting a value of 27 mg/cm² to another lab reporting a value of 75 mg/cm² for the same 45 mg/cm² sample (Figure 21). After the analysis of the initial Round Robin data Lab H realized that their temperature control was 2°C below the required 30°C test temperature and caused systematically low test results. After Lab H corrected their temperature control, the Round Robin results improved slightly from a standard deviation of 45 mg/cm² ± 21.88 in the initial study to 45 mg/cm² ± 18.07 in the revised study, but the results are still significantly outside the error bands stated in the ASTM G67 test standard.

**ASTM G67 RECOMMENDATIONS**

Variability associated with destructive ASTM G67 testing can be reduced by implementing the following procedural changes:

1. Standardize sample cleaning to require use of an ultrasonic bath.
2. Require use of a calibrated temperature sensor and data logger to record the temperature of the oven or water bath for the entire duration of the 24-hour sample exposure.
3. Periodic quality control testing of ALCOA standardized test samples.
4. Measurement of sample dimensions post exposure to develop correlation with material microstructure.
5. Retest areas where the delta between run 1 and run 2 is over 10% of the averaged value.

**Nondestructive Sensitization Testing**

The Degree of Sensitization (DoS) Probe was developed over a five-year period with funding from: NAVSEA 21, NAVSEA 05, ONR SWAMPWORKS, ONR RTT, ONR STTR, and CIT and technical oversight from NAVSEA 05 and NSWCCD. The DoS Probe is an environmentally robust, portable device...
Quantitative Nondestructive 5XXX Aluminum Material Assessments to Reduce Total Ownership Cost

capable of attaching to the complex geometries of ship superstructures in both deck and bulkhead orientations. After completing a 20-minute electrochemical measurement, the DoS Probe displays a numerical result equivalent to the ASTM G67 mass loss value. DoS Probe measurements can be used by repair facilities to assist in maintenance decisions and during planning activities to produce more accurate work plans and reduce total ownership costs. Specifically, in-service material with DoS values greater than 60 mg/cm\(^2\) cannot be welded and must be replaced, material with DoS values between 30 mg/cm\(^2\) and 60 mg/cm\(^2\) can be welded but require a specific cold work treatment, material with DoS values between 30 mg/cm\(^2\) and 20 mg/cm\(^2\) can be weld-repaired using critical weld requirements, and material with a DoS value of less than 20 mg/cm\(^2\) can undergo a normal weld repair.

**DOs Probe Mechanical Design**

The DoS Probe is a field portable electrochemical cell constructed of machined chemically resistant high-temperature PVC, it is fastened with 316 stainless steel fasteners and has an anodized aluminum bottom plate for added durability. The removable tank at the top of the probe contains an internal counter electrode and is filled with a diluted nitric acid solution. In the center portion of the probe is the “probe body” which contains the measurement chamber, a removable reference electrode, a stainless steel spring at the bottom of the probe that provides an electrical connection to the substrate (working electrode), and a spring mounted RTD to accurately measure the temperature of the measurement surface. In front of the measurement chamber is an electronics housing that contains the DoS Probe printed circuit board (PCB) and external connection ports for the Reference Electrode (RE), Counter Electrode (CE), Working Electrode (WE), and circuit ground (GND) as well as the four DoS Probe cable connection ports. At the base of the probe there is a surface heater capable of controlling the temperature of the measurement surface and three pneumatically controlled suction cups used to attach the probe to the structure of interest. The DoS Probe case contains the temperature control box, surface perpetration equipment, spare parts, tools, and additional electrolyte tanks.

**DOs Probe Electrical Design**

The DoS Probe electrical design consists of a limited functionality potentiostat capable of holding a constant potential between the reference electrode and the material being measured (working electrode). This potential difference accelerates the dissolution of intergranular beta phase in the controlled measurement area and the DoS Probe electronics measure, and records the associated current flowing from the material to the counter electrode.

**Electrochemical Methodology**

The DoS Probe is able to accurately quantify DoS by intentionally exposing the area of interest to a very localized and controlled corrosive environment and measuring the current associated with the dissolution of the intergranular beta phase in that controlled area (Figure 23).

Specifically, the DoS Probe exposes the surface to a 2% nitric acid solution that preferentially dissolves grain boundary beta phase if present. A temperature control system controls the temperature of the measurement surface to 50°C to speed up beta phase dissolution and provide a uniform temperature for all DoS Probe readings. The DoS Probe potentiostat circuit holds a specific voltage between the reference electrode and working electrode and measures the current between the counter electrode and the working electrode for the duration of the 20-minute measurement. If the area being tested is highly sensitized, a significant amount of intergranular beta phase will dissolve, resulting in higher measured currents. Similarly, if the area being tested is not sensitized, there will be very little intergranular beta phase to dissolve and measured currents will be small.

The total measured current consists of two parts: 1) The current associated with the dissolution of beta-phase precipitates (Figure 25). 2) The current associated with increased surface area resulting from grain fallout. Because the DoS probe controller maintains a constant potential voltage when the surface area of the sample increases, it decreases the resistance through the electrochemical cell and the measured current increases according to Ohm’s Law: Voltage (V) = Current (I) * Resistance (R).

The two components of the total measured current correlate to the two components that account for mass loss in ASTM G67.
testing. The current associated with the electrochemical dissolution of beta-phase precipitates correlates with the mass loss of just the beta phase and the increased current as a result of increased surface area resulting from grain fallout correlates with the additional mass loss in ASTM G67 attributed to grain fallout.

**DOS PROBE CURRENT CURVES**

The DoS Probe records the measured current during the 20-minute measurement, and when plotted the curves resemble those shown in Figure 23. Each current curve is actually the sum of two separate curves.

Al-Mg matrix material will passivate and form an oxide layer during the DoS Probe measurement. This oxide layer will increase the resistance of the electrochemical circuit and actually result in a decrease in current throughout the measurement (Figure 26).

When beta-phase precipitates are present during a DoS Probe measurement, the dissolution of the beta-phase results in a current increase. The current also increases as intergranular corrosion occurs and the surface area increases as grains fall out (Figure 27).

Sensitized samples will also form an oxide layer in the area directly above each Al-Mg solid solution grain during the DoS Probe measurement. The increased resistance associated with this oxide layer will be negated by the decreases in resistance associated with the increasing surface area as a result of IGC and the increased current from beta phase dissolution.

Therefore, most sensitized DoS Probe current measurements will resemble the sum of the two previous graphs where the decrease in current is only observed in very beginning of the measurement before the intergranular corrosion has started (Figure 28).

**DOS PROBE LABORATORY VALIDATION TESTING**

During the DoS Probe research and development program over 200 Al 5456 ASTM G67 core samples were gathered from CG fleet. The use of fleet material in the development and validation of the DoS Probe methodology was important because it ensured a sample set with naturally sensitized material from a wide range of potential environmental exposures, manufactured 30+ years ago using the technology and processes of that time, multiple manufacturers, varying compositions, different material lots, and the full range of grain sizes and microstructures. Each sample had been tested by ALCOA's ASTM G67 test lab so statistically significant correlations could be made between the DoS Probe measured current and the ASTM G67 mass loss results.

After using these samples to test and refine the DoS Probe methodology, a formal laboratory validation test consisting of 136 runs on 40 different samples was conducted.

When looking at all of the current graphs on the same plot, it became clear that you could visually distinguish sensitized recrystallized material from sensitized unrecrystallized material.
An algorithm was developed to categorize each DoS Probe reading into one of the identified groups and a specific calibration curve was developed for each group. The ability to categorize DoS Probe measurements into groups with specific calibration curves allowed a nondestructive methodology to be developed that could mimic the bias inherent in the ASTM G67 destructive test. With the algorithm and corresponding calibration curves implemented, the 136 test, 40 fleet sample laboratory validation testing yielded a 94% correct binning rate with an $R^2$ value of 0.96.

Additional analysis of the 136 DoS Probe tests included a Run A vs. Run B analysis to examine the repeatability of measurements on the same sample. DoS Probe measurements on the same sample when plotted against one another exhibited a $0.99 R^2$ correlation coefficient. An analysis of the Run 1 vs. Run 2 values for the corresponding ASTM G67 tests competed on the same samples used in the laboratory testing exhibited a 0.89 correlation coefficient (Figure 33). Therefore, during this testing the DoS Probe exhibited improved same sample measurement repeatability than ASTM G67 testing.

**DOS PROBE FIELD VALIDATION TESTING**

After successfully completing laboratory validation testing, the DoS Probe was fleet-tested on two CGs currently undergoing repair. It was a live test at the BAE shipyard in Norfolk with representatives from NAVSEA 05 present. There were five locations on the two ships where destructive ASTM G67 testing had been performed and the DoS Probe was to take a reading next to the ASTM G67 sample area and the results were to be compared. The DoS Probe was able to successfully bin all five of the readings taken during the first field validation.

The DoS Probe then began use supporting measurements in support of CG modernization planning activates and over 800 readings were taken on 8 CGs in the 2013-2014 time period. As part of the quality control program all DoS Probe readings are uploaded to an online database where they are reviewed daily for any anomalies and repeated if necessary to ensure the most accurate measurement data. In conjunction with the 800 DoS Probe tests during the 2013-2014 year of field testing, 44 ASTM G67 samples were taken, and in mid-2014 a one-year DoS Probe performance review was held where the ASTM G67 test results were compared to the DoS Probe readings. The DoS Probe maintained an approximate 90% successful binning rate correlation with ASTM G67 testing across four different laboratories and the DoS Probe remained approved for use by NAVSEA 05.
**DOS Probe Refinements**

There is an ongoing effort to continuously add samples to the DoS Probe calibration dataset to make the nondestructive protocol as robust as possible. Currently, additional midrange samples and large gauge samples of all three microstructures are being tested to continue to improve and refine the correlation between the DoS Probe and the ASTM G67 destructive test.

**DOS Probe Costs**

The DoS Probe manufacturer charges a per measurement fee that includes the following components: electrolyte canister(s), technical support, nightly data review, and minor maintenance and repairs. The low volume per measurement cost is $995 per measurement. A 20% discount per measurement is applied when a large number of measurements are contracted, for example, to support extensive use during CG 2-4-6 phased modernization across 11 ships. Additional testing costs associated with DoS Probe testing include paint removal and replacement and technician labor associated with usage of the equipment. It is estimated that coating removal and replacement and technician labor add approximately $200 to the cost of each DoS Probe measurement.

**Conclusion**

The use of quantitative nondestructive degree of sensitization testing to develop more accurate work specifications resulting in predictable execution of maintenance, modernization, and repair availabilities and to offset costs associated with destructive testing will significantly reduce the total ownership costs associated with vessels containing significant aluminum structural components.

**REFERENCES**


**Author Biography**

**Ryan Dunn** is co-founder and chief executive officer of ElectraWatch, Inc. Mr. Dunn holds a B.A. in mathematics from Skidmore College, a B.E. in electrical engineering from Dartmouth College, and a Master’s of Engineering Management from Dartmouth College.