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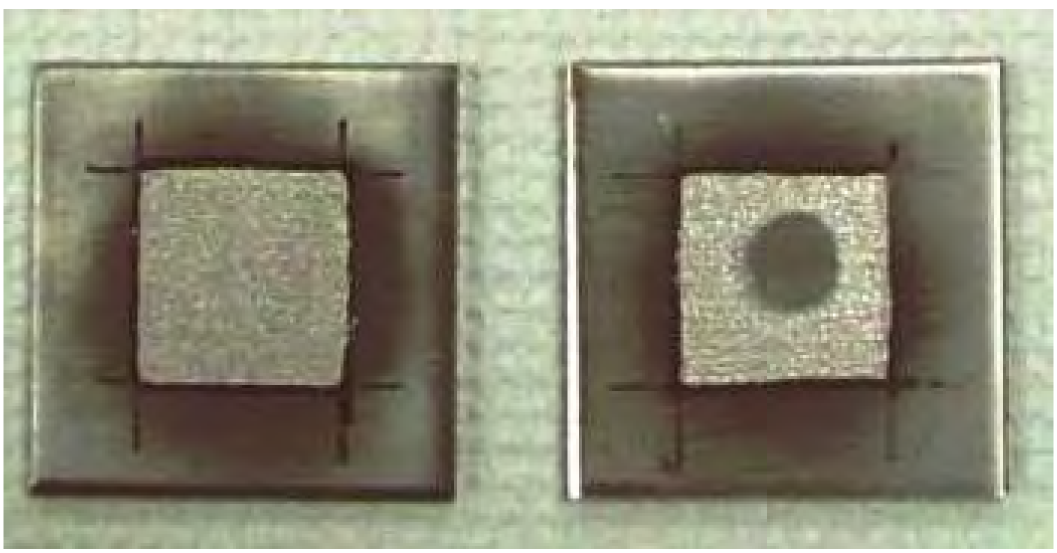
CAVITATION EROSION OF ELECTRO SPARK DEPOSITED NITINOL VS. STELLITE® ALLOY ON STAINLESS STEEL SUBSTRATE



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14. ABSTRACT

The Naval Facilities Engineering and Expeditionary Warfare Center (NAVFAC EXWC) was tasked with determining the feasibility of combining Nitinol (NiTi) superelastic metal alloy with ElectroSpark Deposition (ESD) technology to increase the cavitation erosion resistance of fluid machinery utilized by the Department of Defense (DOD). EXWC examined several industrial pump materials and compared their relative cavitation erosion resistance. The ESD'd Nitinol was also examined by Differential Scanning Calorimetry (DSC) and Dilatometry (DIL) for changes in its thermo-mechanical properties due to ESD. Microscopy was conducted to examine the metallurgical bond established by the ESD process, and its subsequent erosion, per ASTM G32. As a comparison, a known erosion-resistant, weld-friendly alloy called Stellite 6® was ESD'd and its cavitation erosion resistance compared to that of Nitinol.

15. SUBJECT TERMS

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EXECUTIVE SUMMARY

The Naval Facilities Engineering and Expeditionary Warfare Center (NAVFAC EXWC) was tasked by the Corrosion Prevention & Control (CPC) program office of the Office of Secretary of Defense (OSD) to examine a unique treatment technology known as "Electro Spark Deposition" or "ESD", in partnership with a special superelastic metal alloy called Nitinol. The purpose of the examination is to determine the feasibility of using this combination of technologies to increase the cavitation erosion resistance of fluid machinery utilized by the Department of Defense (DOD). Nitinol is known for its superior cavitation erosion resistance, but it is a relatively expensive alloy from which to fabricate entire components. In theory, ESDing this costly alloy onto cheaper, less resistant metals for seawater immersion use could greatly improve the cavitation resistance at a much lower cost.

EXWC conducted a laboratory study in which several industrial pump materials were tested and compared for their relative cavitation erosion resistance. Superelastic Nitinol was tested before and after being electro spark deposited onto stainless steel substrate. The ESD process was conducted both in-house and by a vendor specialized in the technique. The ESD'd Nitinol was also examined by Differential Scanning Calorimetry (DSC) and Dilatometry (DIL) for changes in its thermo-mechanical properties due to ESD. Microscopy was conducted to examine the metallurgical bond established by the ESD process. As a comparison, a known erosion resistant, weld-friendly alloy called Stellite 6® was ESD'd and its cavitation erosion resistance compared to that of Nitinol.

The evaluation revealed that Nitinol is a poor candidate for electro spark deposition. The material was difficult to apply to 300 series stainless steel and, more importantly, appears to lose its martensitic-austenitic phase transition when ESD processed. This solid-solid phase transition is believed to impart Nitinol with its superior erosion resistance properties. The alternative alloy tested, Stellite 6®, performed extremely well in the cavitation erosion tests, both before and after the ESD process.

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ACRONYMS AND ABBREVIATIONS

ASTM	American Standards & Test Methods
Cr	Chromium
Cu	Copper
ESD	Electro Spark Deposition
Fe	Iron
HB	Brinnell Hardness
in	Inch
kgf	Kilogram-force
ksi	Kilo pounds per square inch, 1000 pound unit
min	Minute
mm	Millimeter
Mn	Manganese
Mo	Molybdenum
N	Nitrogen
NAVFAC EXWC	Naval Facilities Engineering & Expeditionary Warfare Center
Ni	Nickel
NiTi	Nitinol alloy
NPSHA	Net-Positive Suction Head Available
OSD (AT&L)	Office of the Secretary of Defense Acquisition, Technology and Logistics, Department of Defense
ppb	Parts per billion
psi	Pounds per square inch
r_e	Erosion Rate
R_e	Erosion Resistance

Si	Silicon
SS	Stainless Steel
µF	Microfarad – a measure of electric capacitance
µg/L	Micrograms per Liter
µm	Micrometer
°C	Degrees Celsius (Centigrade)
®	Registered brand name

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1.0 INTRODUCTION

The Naval Facilities Engineering and Expeditionary Warfare Center (NAVFAC EXWC) was tasked by the Corrosion Prevention & Control (CPC) program office of the Office of Secretary of Defense (OSD) to examine a unique treatment technology known as "Electro Spark Deposition" or ESD, in partnership with a special superelastic metal alloy called Nitinol. The purpose of the examination is to determine the feasibility of using this combination of technologies to increase the cavitation erosion resistance of fluid machinery utilized by the Department of Defense (DOD).

EXWC conducted a laboratory study in which several industrial pump materials were tested and compared for their relative cavitation erosion resistance. Superelastic Nitinol was tested before and after being electro spark deposited onto stainless steel substrate. The ESD process was conducted both in-house and by a vendor specialized in the technique. The ESD'd Nitinol was also examined by Differential Scanning Calorimetry (DSC) and Dilatometry (DIL) for changes in its thermo-mechanical properties due to ESD. Microscopy was conducted to examine the visual metallurgical bond established by the ESD process. As a comparison, a known erosion resistant, weld-friendly alloy called Stellite 6® was ESD'd and its cavitation erosion resistance compared to that of Nitinol.

The evaluation revealed that Nitinol is a poor candidate for electro spark deposition. The material appears to lose its martensitic-austenitic phase transition when ESD processed. The transition is believed to impart Nitinol with its superior erosion resistance properties. The alternative alloy Stellite 6® performed extremely well in the cavitation erosion tests, both before and after the ESD process.

2.0 BACKGROUND

2.1 Cavitation Erosion

Cavitation erosion is a common problem that afflicts fluid machinery essential to the proper functioning of many DOD assets. Pump impellers, water turbines, hydraulic torque converters, and similar seawater components aboard Navy ships and at the waterfront are subject to severely damage or destruction due to cavitation erosion, leading to shortened service life and poor performance. Figure 2-1 shows an example of an impeller damaged by cavitation erosion. The consequences of cavitation erosion damage range from system downtime and repair to sudden catastrophic failure of the entire system.

Figure 2-1 Example of Cavitation Erosion on a Pump Impeller

The normal movement of components in fluid machinery often contributes to the phenomenon known as cavitation erosion. High-speed fluids passing around, for example, a moving impeller, cause turbulence and generate tiny “vacuum” bubbles. Intense localized pressure of these bubbles at the component’s surface causes their collapse. High-energy shock waves are formed from the collapse and carry enough energy to rip minute pieces of metal from the component’s surface. Additionally, vibrations from the now damaged moving part may transmit to nearby components such as mechanical seals and bearings, causing further damage.

Attempting to reduce cavitation erosion by decreasing operating speeds or frequency is detrimental to system performance. Fabricating entire components from highly cavitation resistant materials is often cost-prohibitive. Alternately, a more economically viable option is to incorporate highly cavitation-resistance materials onto more susceptible machine components as a “coating”. By coating, we are referring to the application of metal onto metal, rather than the traditional polymeric coatings, which are too soft to provide any long-term protection from cavitation erosion. [1]

Of particular interest to the Navy are pump materials resistant to seawater. Common alloys utilized in seawater pumps are listed in Table 2-1, along with their relative corrosion potentials in flowing seawater, and relative costs:

Table 2-1 Relative Ratings for Common Seawater Pump Alloys

Alloy	**Relative Galvanic Corrosion Resistance in Flowing Seawater** [2] [3] **1 = Lowest, 6= Highest (Stagnant Seawater)**	**Relative Cavitation Erosion Resistance** [2] [3] **(1 = Lowest, 6= Highest)**	**Relative Cost** [3] **(1 = Lowest, 6 = Highest)**
Standard Austenitic Stainless Steel – 300 Series	2 for SS-304 (1 in stagnant) 3 for SS-316L (1 in stagnant)	1-2	1-2
Nickel Aluminum Bronze	1-2	3-4	3
Super Austenitic Stainless with 6% Molybdenum	4-5	3-4	5
Duplex Stainless	4-5	3-4	4
Nickel-based Inconel and Hastelloy	6	6	6
Titanium Alloys	6	5	6

Desalination systems used on Navy ships and DOD expeditionary are known to be subjected to especially severe environments due to the high chloride, high temperature conditions. In the past, austenitic stainless steels in the ASTM 300 series tended to be the material of choice in desalination equipment. In cases in which higher corrosion resistance has been needed, austenitic 6% Molybdenum grades and, more recently, Duplex 2205 and other duplex steels have emerged as optimal materials in many situations. Additionally, increasingly stringent state and federal discharge limits into littoral waters has necessitated a shift away from alloys that leach contaminant metals such as copper. The California Water Board has set the current maximum copper discharge limit at the Port of Hueneme to 2.7-µg/L or ppb. [4] EXWC's Seawater Desalination Test Facility (SDTF) has been forced to eliminate copper alloys, such as nickel aluminum bronze or CDA 958, from its equipment and piping.

2.2 Electro Spark Deposition

The technique known as "Electro Spark Deposition" or "ESD" is an established technology utilized by the aerospace industry. In ESD, short-duration electrical pulses are discharged at controlled energy levels through a spinning metal electrode. The electrode is made of the desired deposit alloy. The tip of the electrode melts as it spins and contacts the substrate material, depositing itself onto the substrate (Figure 2-2). Rapid solidification produces nano-structures with unique tribological and metallurgical characteristics. When utilizing appropriate fusible materials, the resulting deposit-substrate interface becomes a true metallurgical bond up to 0.5 μm deep.

Figure 2-2 Close up of ESD Applicator Electrode at Substrate Surface

The low total heat input of ESD allows the bulk of the surrounding substrate material to remain near ambient temperature, avoiding heat zones that can damage the substrate. This feature separates ESD from regular TIG welding, where thermal stresses to the substrate are a given. Another metal deposition technique, thermal spray, operates at high temperatures that can deform substrates and contaminate their microstructure with oxygen, vacancies, and other foreign substances. Welding also tends to deposit an uncontrolled and overly thick metal layer onto the substrate, whereas ESD typically deposits coatings at 0.001 to 0.003-inch per pass. Thicker layers are built up incrementally, as desired.

Nitinol alloy, the focus of this study, is a truly unique material possessing what is known as "superelastic" or "shape memory" properties. The name "Nitinol" is derived from its components and original developer: "Ni" for Nickel, "Ti" for Titanium, and "Nol" for the Naval Ordnance Laboratory (NOL). [5] In 1961, researchers at NOL in Maryland discovered, by accident, that their nickel titanium alloy could spring back to its original form after being bent out of shape then heated. Hence, "shape memory".

Shape memory alloys are able to undergo reversible strain up to 10% and revert to their original shape, without permanent deformation. These alloys are strong and tough. They have the ability to endure tremendous amounts of strain, requiring immense effort to cut and allowing them to absorb energy from shock waves in a fluid. These attributes give Nitinol its outstanding cavitation erosion resistance, 20 to 60 times that of stainless steel. [6] [7]

Nitinol contains nominally 54.5 to 57% by weight Nickel, with the balance being Titanium and trace elements. Through a solid-solid "martensitic-austenitic" phase transformation, the material goes from one crystal structure (monoclinic) to another (cubic) upon heating, and the reverse of this transformation upon cooling. [8] Below the transition, Nitinol is in the martensitic phase and can be bent into various shapes. Above the transition, Nitinol is in the austenite phase and reverts to its "parent" shape. Nitinol is considered superelastic if the solid-solid transition is below the use temperature of the material. Nitinol is considered shape memory if the transition is above the use temperature. The transition temperature range is dependent on the exact composition of the alloy as well as its processing history.

Nitinol is an expensive alloy, and it is generally not cost effective to machine entire NiTi components, other than for medical devices, and/or small items. Because Nitinol is difficult to weld, cladding is not an option. However, thinner layers of Nitinol or other high-CE-resistance materials coated on traditional materials by ESD could be cost effective if they impart their desirable properties to less expensive substrate metals.

3.0 TESTING PROTOCOL

3.1 Materials

Materials procured for the laboratory study are presented in Table 3-1 below. In addition to Nitinol and Stellite 6®, other materials were selected based upon their frequent usage in marine environments, seawater pumps in particular:

Table 3-1 Materials Selected for Testing

Name	Form / Size	Composition	Information	Providing Vendor
Nitinol [9]	Wire, 0.125-in diameter	Straight Annealed, Ni 55.93%, Ti=Balance, C<0.05%, O<0.05%, Other elements total <0.2%	Transition Active Af=17.1°C Thus material is fully austenitic > 17.1°C (room temperature)	Johnson Matthey
Nitinol [10]	Coupons, 2-in by 2-in by 0.059" thick	Flat Annealed, Ni 55.89%, Ti=Balance, C<0.05%, O<0.05%, Other elements total <0.2%	Transition Active Af=6.9°C. Thus material is fully austenitic > 6.9°C (room temperature)	Johnson Matthey
SS-304 [11]	Coupons, 2-in by 2-in by 0.125-in thick	Cold-Rolled Annealed, Cr 18%, Ni 8.1%, Mn 1.2%, Si 0.5%, Fe Balance. Other elements total <0.2%	Standard stainless steel, offering a combination of corrosion resistance, workability, and low cost. Vulnerable to stress corrosion cracking. More susceptible to pitting corrosion than SS-316.	Online Metals
SS-316L [12]	Coupons, 2-in by 2-in by 0.125-in thick	Cold-Rolled Annealed, Cr 16.63%, Ni 10.07%, Mo 2.06%, Mn 1.47%, Cu 0.42%, Si 0.24%, Fe Balance. Other elements total <0.1%	Standard stainless steel, offering a combination of corrosion resistance, workability, and medium cost. Similar vulnerability to stress corrosion cracking as SS-304. Less susceptible to pitting than SS-304, but more than SS-2205. SS-316L differs from SS-304 in that it contains Molybdenum as well as a greater proportion of Nickel and a lower proportion of carbon. The lower carbon content of SS-316L allows lower carbide precipitation for welding.	Online Metals
SS-2205 Duplex Stainless [13]	Coupons, 2-in by 2-in by 0.125-in thick	Coil, Cr 22.4%, Ni 5.79%, Mo 3.13%, Mn 1.47%, Si 0.42%, N 0.17%, Fe Balance. Other elements <0.1%	Tough, highly corrosion-resistant alloy with more Molybdenum and Chromium than either SS-304 or SS-316. Widespread use in marine applications. Less easily machined & more resistant to both pitting corrosion and stress corrosion cracking than SS-304 and 316.	Metal Samples Company

Name	Form / Size	Composition	Information	Providing Vendor
254SMO [14] **6% Molybdenum**	Coupons, 2-in by 2-in by 0.125-in thick	Coil, Cr 19.9%, Ni 17.9%, Mo 6.1%, Cu 0.73%, Mn 0.51%, Si 0.31%, N 0.2%, Fe Balance Other elements ,0.1%	Used for "marine hardware, valves, impellers, and other pump parts in contact with seawater. High corrosion and erosion resistance. Superior resistance to multiple forms of corrosion: pitting, crevice, and stress corrosion cracking, compared to typical stainless steels like SS-316.	Metal Samples Company
CDA958 (C95800) [15] **Nickel Aluminum Bronze (majority Copper)**	Coupons, 2-in by 2-in by 0.125-in thick	Cast, Cu 81.93%, Ni 4.42%, Al 9.05%, Fe 4.14%, Mn 0.93% Other elements <0.1%	Used for "marine hardware, valves, impellers, and other pump parts in contact with seawater. High corrosion and erosion resistance.	Metal Samples Company
Stellite® 6B [16] [17]	Coupons, 2-inch diameter rounds, 0.25-inch thick	Stellite 6B Cast Rounds, Cobalt Base, Cr 30%, W 5%, C 0.9-1.4% with trace Ni, Fe, Si, Mn, and Mo.	Cobalt-based alloy with excellent resistance to wear, galling, and corrosion. Retains its properties at high temperatures. Can be turned with carbide tools. Used in valve seats, pump shafts and bearings, erosion shields. Corrodes by pitting mechanism, not by general mass loss in seawater and chloride solutions.	WeldTool Technologies Inc.
Stellite® 6 [16]	Wire, 3/32-inch diameter 1/8-inch diameter	Stellite 6 Electrodes, Cobalt Base, Cr 30%, W 5%, C 0.9-1.4% with trace Ni, Fe, Si, Mn, and Mo	Same as above.	Surface Treatment Technologies

3.2 ESD Process

EXWC initially sent out stainless steel coupons to a commercial vendor to be ESD'd with Nitinol (wire electrode), also supplied by the Navy. This vendor, Advanced Surfaces and Processes (ASAP) was previously a commercial partner to Pacific Northwest National Laboratories, an inventor of electro spark deposition methodology and apparatus. [18] ASAP now terms their process "nano-fusion" and states that it is generations advanced from the original ESD process.

At the time this project was conducted, vendor availability to provide ESD services was extremely limited. This combined with vendor inability to supply required samples led EXWC to acquire its own hand-held ESD equipment from Surface Treatment Technologies, Inc. This allowed us to prepare additional coupons with Nitinol and Stellite® according to our requirements. The Spark Depo 300 is a semi-portable unit that can be operated in the field from a mobile cart. It utilizes a standard hand-held electrode applicator and includes shield gas integration capability. A shield gas, similar to that used in welding operations, promotes the formation of a homogeneous metallurgical bond and deposition layer, and supports prevention of impurity formation.

The operating parameters for the ESD overlay applications are listed below:

- Shield Gas = Argon
- Gas Flow Rate = 2-3-Liters/min
- Output Capacitance = 40-µF
- Frequency Setting = 5 (equivalent to approximately 360-Hz)
- Voltage = Low – 150 Volt

3.3 Differential Scanning Calorimetry and Dilatometry Analysis

EXWC sent samples of the superelastic Nitinol material, the SS-304 substrate material, and Nitinol ESD'd onto SS-304 substrate to TA Instruments for Differential Scanning Calorimetry (DSC) analysis. TA Instruments is a world leader in the manufacture of thermal analysis instrumentation. They evaluated the Nitinol's solid-solid austenitic/martensitic thermal transition before and after ESDing using their Tzero™ Q2000 DSC, and referencing ASTM F2004-05 "Standard Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis". [19]

DSC is an analytical technique for measuring a material's heat flow as a function of time and temperature. Heat flow is associated with changes in the material's structure. Measurements provide qualitative and quantitative information about physical and chemical changes in materials.

TA instruments ran the materials from well below zero Celsius (-50°C) to nearly 150°C at 10° C./in and at 20°C/min. to properly capture any phase changes in the materials. The SS-304 substrate material was used in the reference pan of the instrument to improve sensitivity for capturing very small transitions measured in milliwatts.

3.4 Cavitation Erosion Testing

Cavitation-erosion (CE) tests were conducted according to ASTM G32-09 "*Standard Test Method for Cavitation Erosion Using Vibratory Apparatus, Note 1 Indirect Method*" on metal coupons of the procured materials, and on Nitinol and Stellite® wire that was ESD'd onto stainless 304 coupons (see Section 3.1). [20] A Sonics® CV334 probe with 0.5-inch (13-mm) diameter replaceable titanium Ti-6Al-4V tip was used as the vibratory apparatus, along with a Sonics® Vibra-Cell, VC-505 processor to control duration, frequency, and amplitude of the cavitating vibratory action. An Aldrich DigiTrol II temperature probe and monitor were used to monitor the water temperature in which the sample was immersed during testing. A Julabo F250 water chiller with attached copper cooling coil was used to regulate the beaker water temperature. Because of noise generated during the testing by the cavitation probe, a Sonics® sound booth was also utilized, allowing the operator to work nearby without needing additional personal protective equipment.

The operating parameters for all of the tests are listed below:

- Probe Vibrating Frequency = 20-kHz [21]
- Probe Vibrating Amplitude = 50-um [21]
- Gap from probe face to coupon surface = 1.0-mm
- Water Volume = 250-ml deionized water in a 600-ml, 3.25-inch inner diameter glass beaker.
- Water Temperature = Held at 20 – 23°C by the immersed copper coil.
- Chiller Water Temperature = 19°C.

Figure 3-1 Setup for the Laboratory Cavitation Erosion Testing of Coupons

Figure 3-2 Close-up of Cavitation Erosion Test Setup

Prior to testing, each test coupon was ultrasonically cleaned in a Branson 2200 Sonicator with liquid detergent and deionized water for approximately 1 hour. The coupons were subsequently washed with deionized water, immediately dried in a 110°C oven, then cooled and stored in a desiccator. Each coupon as weighed immediately before each test cycle using a Sartorius® Research R160P scale with 0.0001-gm resolution.

Each test coupon was placed inside the plastic holder at the bottom of the 600-ml beaker, and 250 mL of deionized water added. The coiling copper coil was submerged in the water. Then the probe was lowered so that the face of the tip just grazed the coupon surface. The probe's clamp was tightened to lock the probe in place. The 1-mm spacer was then added to the rim of the supporting clamp, raising the probe up precisely 1-mm from the coupon surface when seated on the spacer (Figure 3-3).

Figure 3-3 Close-up of 1-mm Orange Spacer Seated on Rim of Probe Clamp

The coupon was cavitated by the probe for intervals between 45 to 90 minutes. In between CE cycles, the coupon was removed from the test apparatus, rinsed in running water, dried in the oven for approximately 5 minutes, and then cooled in the desiccator for approximately 15 minutes before reweighing. After weighing, the coupon was cavitated again until the cumulative time of these sonic cycles equaled or exceeded approximately 600 minutes total.

After testing of each individual coupon was complete, the spent tip on the probe was replaced with a new tip before testing another coupon. The surface of each used tip was itself eroded from the force of the cavitation environment. Each spent tip was placed in a small zipped plastic bag for later examination.

3.5 Digital 3D Microscopy Examination

A Keyence VHX-2000E high resolution digital microscope (Figure 3-4) with VHZ-Z20W 20-200x lens was used to examine the surface and metallurgical bonding of the ESD'd Nitinol and Stellite® on the steel substrate. The microscope's software is capable of "stitching" together individual images of the surface to provide a 3D profile of the deposited material.

Figure 3-4 ESD Deposit Being Analyzed by Microscope

4.0 RESULTS AND DISCUSSION

4.1 ESD Compatibility with Nitinol

Specimens for testing were prepared in-house and by contractor. ASAP alloyed Nitinol onto several SS-304 coupons supplied by EXWC, using a variety of different parameters in an attempt to optimize the deposited layer. They found 0.001-inch was the maximum thickness of material possible due to the formation of micro-cracks in the "fused Nitinol". ASAP subsequently was able to achieve a deposit thickness of approximately 0.0025-inch by alloying a thin "butter coat" of pure nickel onto the stainless first, followed by successive layers of Nitinol. [22] A cross-sectional photo of the layers is shown in Figure 4-1.

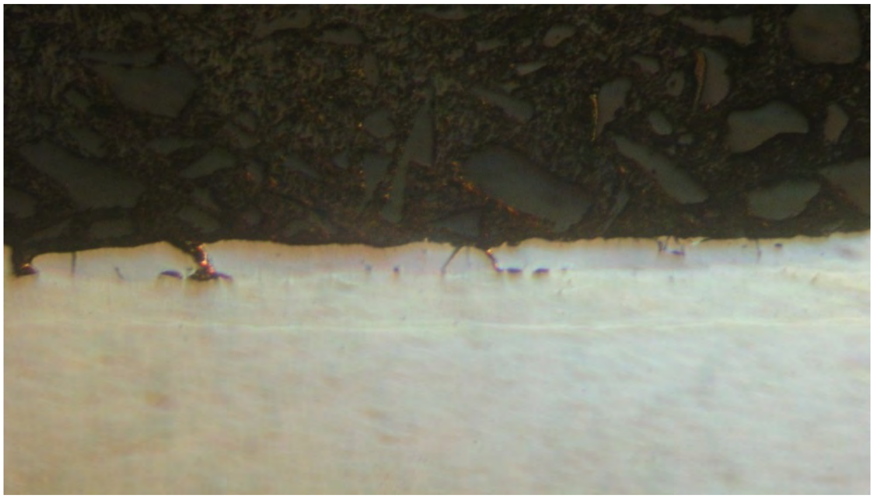

Figure 4-1 Nickel and Nitinol Fused onto SS-304

The team at APAS theorized that Nitinol's extreme hardness, along with high chromium levels in the stainless might be the root cause of the poor compatibility. ASAP also nano-fused Nitinol onto mild steel coupons, which produced somewhat better results, but not good enough to recommend the Nitinol as a viable ESD material. [22]

EXWC subsequently ESD'd additional samples of Nitinol onto SS-304 coupons using the Spark Depo 300 equipment. The material appeared to fuse onto the substrate, although a number of passes were needed to build up any appreciable deposit thickness. Stellite® wire electrode was also ESD'd onto SS-304 for comparison. In Figure 4-2, the coupons on the right side have been cavitation eroded. Using the handheld device required operator skill in applying even layers and the process was very slow.

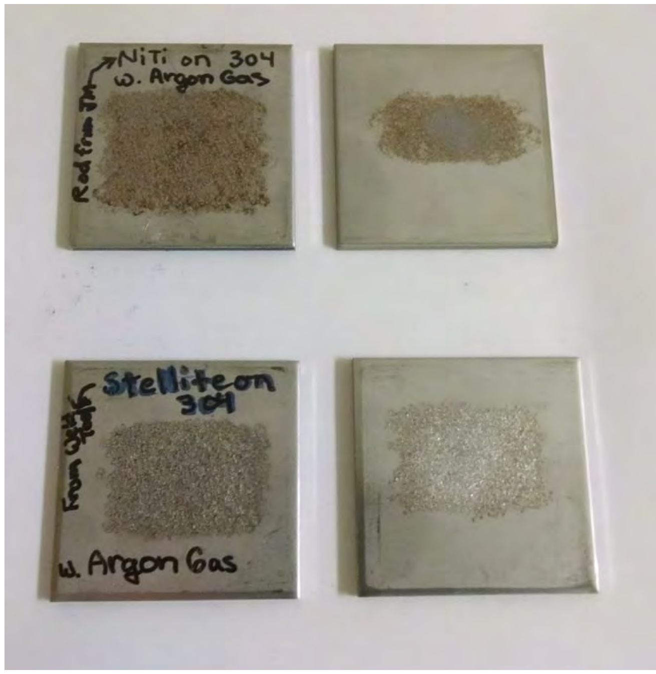

Figure 4-2 Nitinol Vs Stellite ESD'd on SS-304

4.2 DSC and Dilatometry Results

TA Instruments analyzed the SS-304, Nitinol wire, and the ESD'd Nitinol prepared by ASAP. [19] Figure 4-3 clearly shows the DSC phase transition from a martensitic to austenitic state and back as a section of Nitinol wire is heated from -50°C to +100°C, then cooled through the same temperature range. Figure 4-4 shows a first and second heat cycle scan for the Nitinol ESD'd on the stainless substrate. No phase transition is observed, even at an expanded heat flow y-scale. The sensitivity of the DCS should be sufficient to detect a phase transition in the minute amount of Nitinol vs. bulk stainless substrate, if it were to exist.

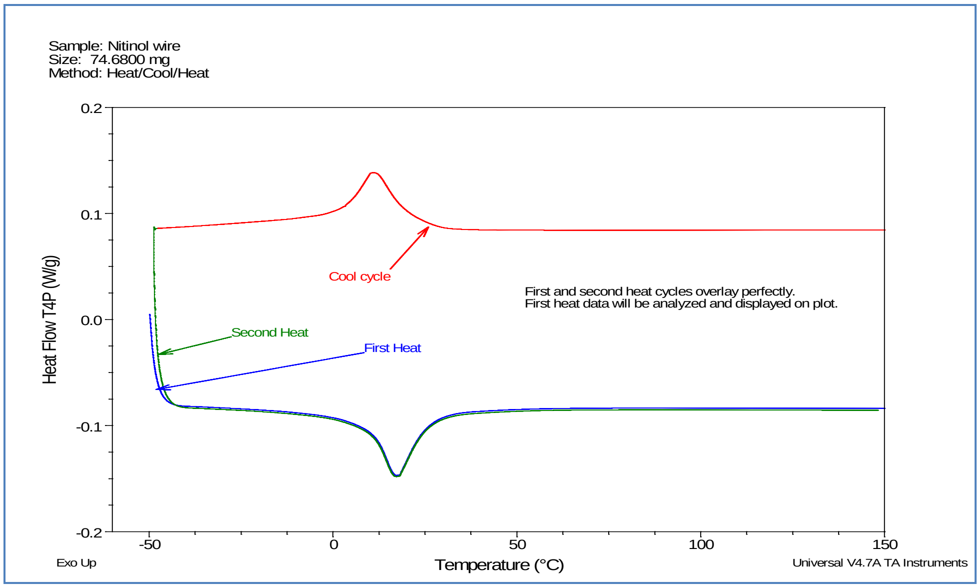

Figure 4-3 DSC Scan of Superelastic Nitinol Wire (Annealed)

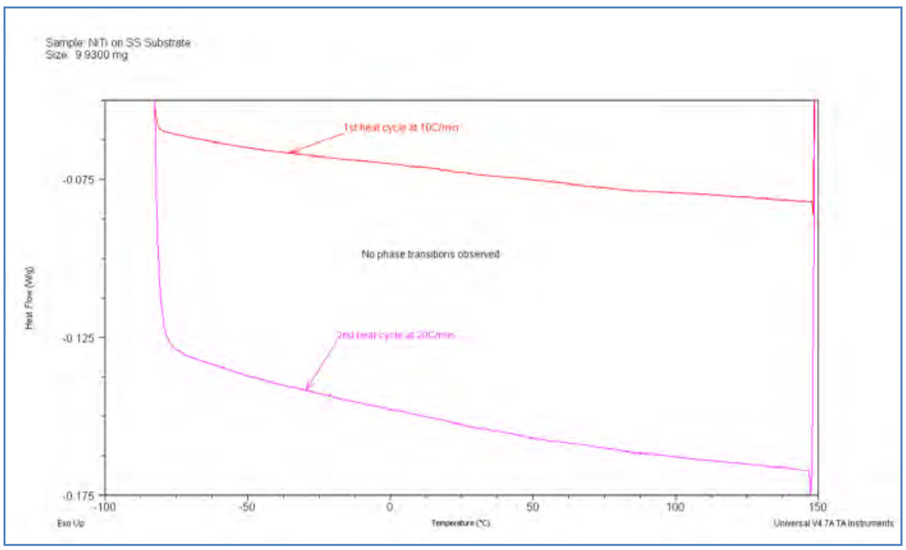

Figure 4-4 DSC Scan of Nitinol Wire ESD'd onto SS-304

Since the layer of Nitinol was very small in comparison to the mass of the underlying stainless substrate, a second test was performed in which a similar mass of only SS-304 was placed into the DSC's reference pan. This served to balance the heat capacity between the reference and sample pans to improve sensitivity. The resulting scan was the same: no phase transition of the ESD'd Nitinol.

Although heat generation is minimal at the macro level during ESDing, there is still substantial heat produced at the "nano" level in the materials fused. EXWC proposes that the ESD process destroys the martensitic-austenitic of the superelastic Nitinol. Since this phase transition is, at least in part, responsible for Nitinol's superior cavitation erosion resistance, it would follow that ESDing the Nitinol would render the material less CE resistant. [7]

Dilatometry was conducted in-house by EXWC personnel to confirm the DSC results. EXWC utilized its Linseis L75 Cryogenic Horizontal Dilatometer for the analysis. Dilatometry (DIL) is the method of choice for highly precise measurement of dimension changes to solids, melts, powders and pastes over a controlled temperature regime. The dimensional change is often expressed as the "thermal coefficient of expansion" or CTE of a material. The solid-solid transition of the superelastic Nitinol is a perfect candidate for evaluation by dilatometry.

Figure 4-5 shows the CTE curves for duplicate sample runs for NiTi rod, ESD'd Nitinol, and SS-304. The transition is clearly detected by the instrument. The SS-304 data was further subtracted from that of the ESD'd Nitinol to seek out any miniscule transitions in the ESD'd material that might be obliterated by the SS-304 baseline. No phase transitions were detected.

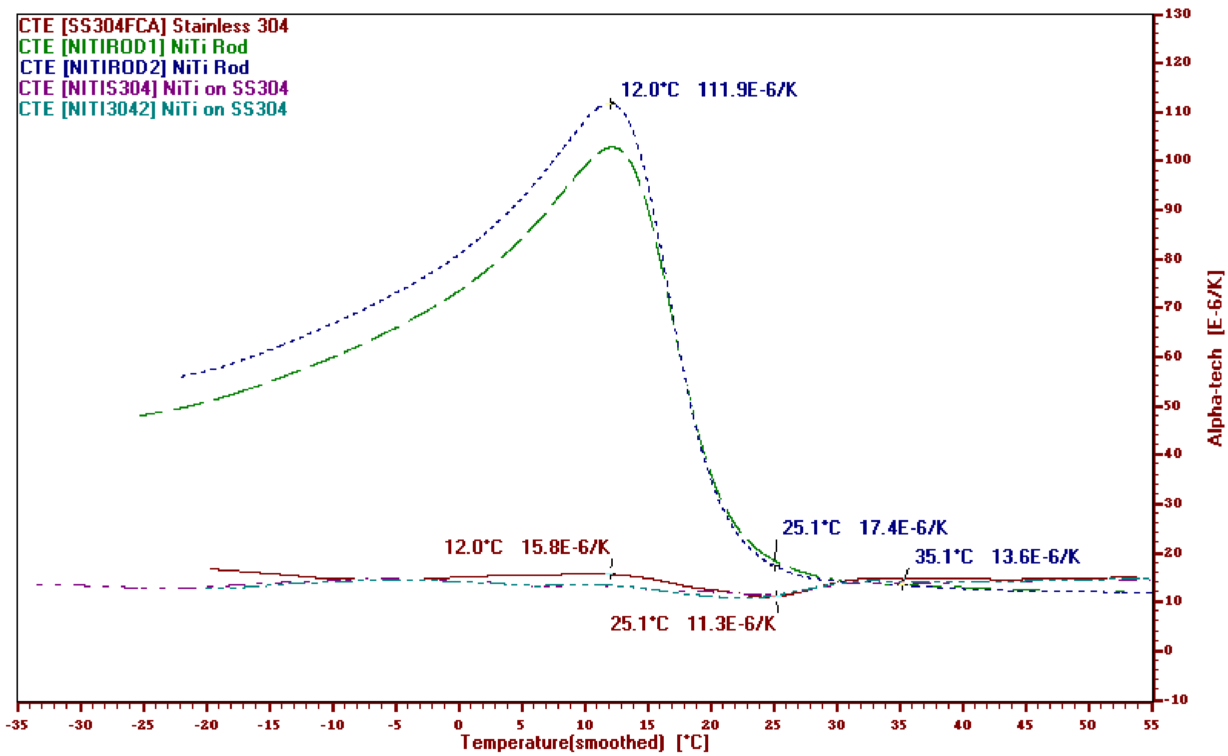

Figure 4-5 Dilatometer Analysis of Nitinol

4.3 Cavitation Erosion Results

The laboratory CE test data for the individual coupons is included in Appendix A. Figure 4-6 shows a compilation of cumulative weight loss for the tested materials. The chart includes data from at least two coupon runs per material. The test setup and procedures described in Section 3 allowed linear and repeatable data to be obtained. Table 4-1 lists the total weight loss at 600-minutes (10-hours) for each tested material. Interpolation was performed for test runs where exactly 600 minutes was not a recorded data point.

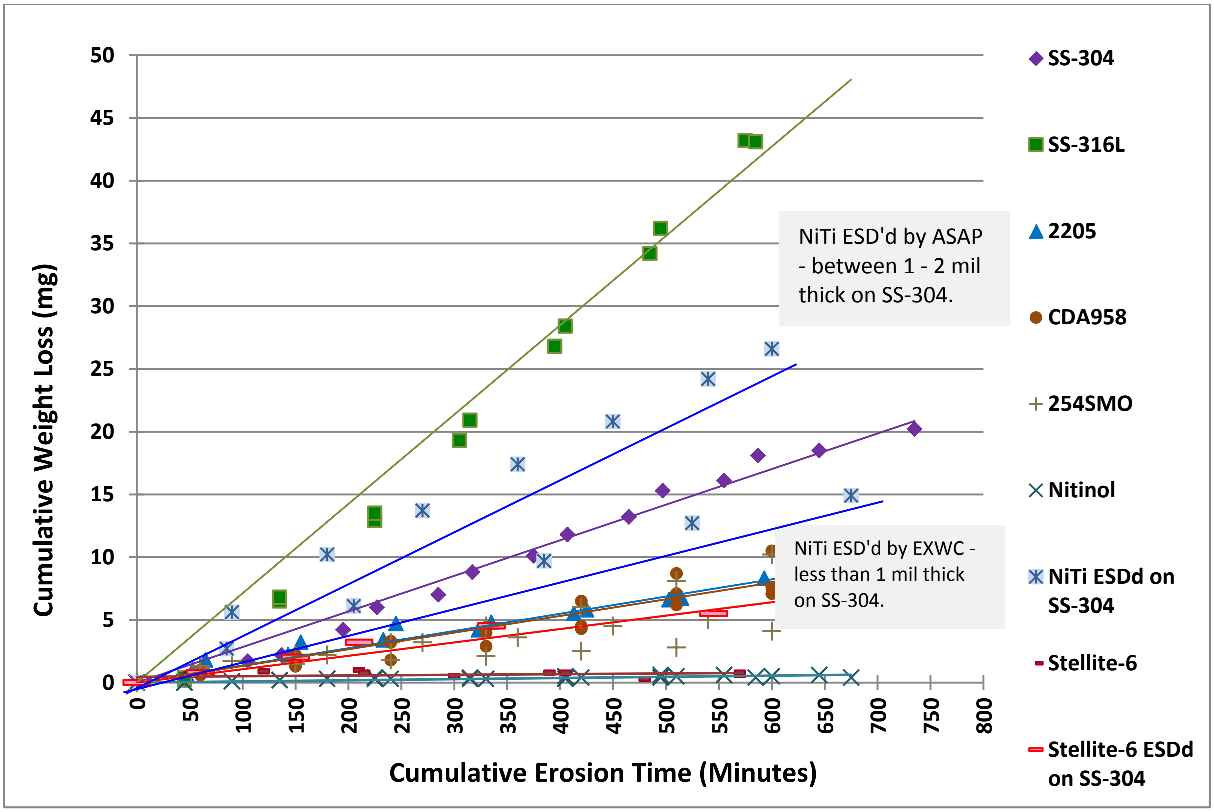

Figure 4-6 Cavitation Erosion Behavior of Various Alloys per ASTM G32

Table 4-1 Total Weight Loss at 600-Minutes

Material	Cumulative Weight Loss (mg)
Nitinol, pure	0.6
Stellite®	1.0
254SMO	6.4
Stellite®, ESD	8.0
CDA958	8.0
SS-2205	8.2
SS-304	17.2
Nitinol, ESD (Avg)	19.6
SS-316L	42.6

Not surprisingly, the coupons of "pure" Nitinol and Stellite® material experienced the least weight loss compared to all other materials. The ESD'd Nitinol performed poorly in comparison. The Nitinol "nano-fused" robotically by APAS experienced significantly more weight loss than the coupons ESD'd by EXWC however. The cause for this is unknown. Figure 4-6 shows the two separate trend lines for these results. The difference between the data sets may be due to a difference in thickness of the deposits. APAS was able to apply a thicker coating due to the primer of nickel on the surface, i.e. 0.002-inch thick, whereas the EXWC coupon deposit was estimated to be only 0.001-inch. One would theorize that the thicker coating would perform better, but if the material has poor ESD properties, a thicker layer might erode faster.

CE testing at the University of Fukui in Japan determined that Nitinol is more erosion-resistant in the martensitic phase than in the austenitic phase. [23] The pure Nitinol received by EXWC is super elastic, meaning it was in its austenitic phase at room temperature. It was CE tested at water temperatures of 20 to 23°C, still in its austenitic phase. To determine if its behavior would change at a martensitic temperature, the CE testing was performed at a water temperature of 5-7°C. No significant changes were observed through 600 minutes (10-hours). To observe appreciable erosion in Nitinol, a test duration of at least 50 hours would likely be required. [23]

The ESD'd Stellite® performed quite well, having the lowest trend line for weight loss, with the exception of the trend lines for the pure Nitinol and Stellite®. Note that the data points for the 6% Mo alloy (254SMO) are significantly more scattered from coupon to coupon than the data for the other alloys (Figure 4-6). The cause for this is unknown. Potentially, there may be homogeneity issues for the tested batch of this material. Its trend line (linear regression) is close to that of the ESD'd Stellite® indicating similar erosion resistance. Figure 4-7 shows "before" and "after" cavitation erosion for SS-304, SS-316, Nitinol, and ESD'd Nitinol. The eroded spot is visually dramatic on all the coupons except pure Nitinol.

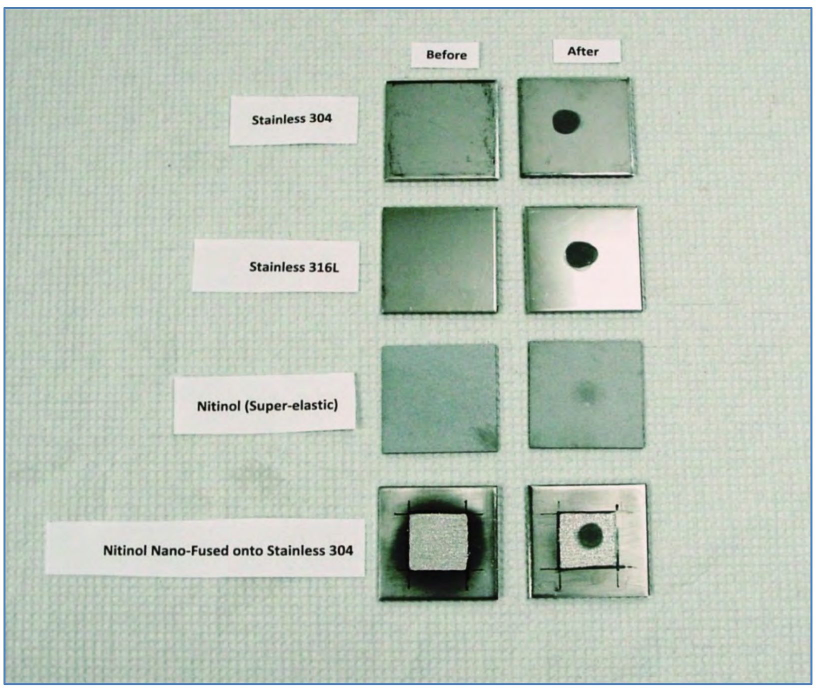

Figure 4-7 Comparison of Alloys – Cavitation Erosion

4.3.1 Comparison of Erosion Rates

The erosion rate r_e for a given coupon over a selected amount of time was calculated as:

$$r_e = \frac{\Delta m}{\Delta t}$$

where Δm represents the change in mass in the coupon over the specified interval Δt. Figure 4-7 displays a graph of erosion rates over time for each alloy. The rates for the tested alloys varied over time. Generally, erosion rates were low at the very beginning of cavitation exposure, then increased rapidly over a cycle or two, then leveled off to a semi-steady state with some variability. This pattern is documented in the literature for ASTM G32 vibratory cavitation of metals. [6] [24] There is an initial period of little to no weight loss called the "incubation period". This period can be quite short, as is seen in Figure 4-7 where several of the alloys experienced a dramatic jump in their erosion rate within 90-minutes. For harder stainless steels and cobalt based alloys, the incubation period can be longer. After some period of time, the erosion rate levels off to a "steady state" of weight loss, hence the linear data seen in Figure 4-6. If a test is prolonged long enough, as seen for SS-304 in Figure 4-7, the rate of weight loss decreases due to surface roughness. [6]

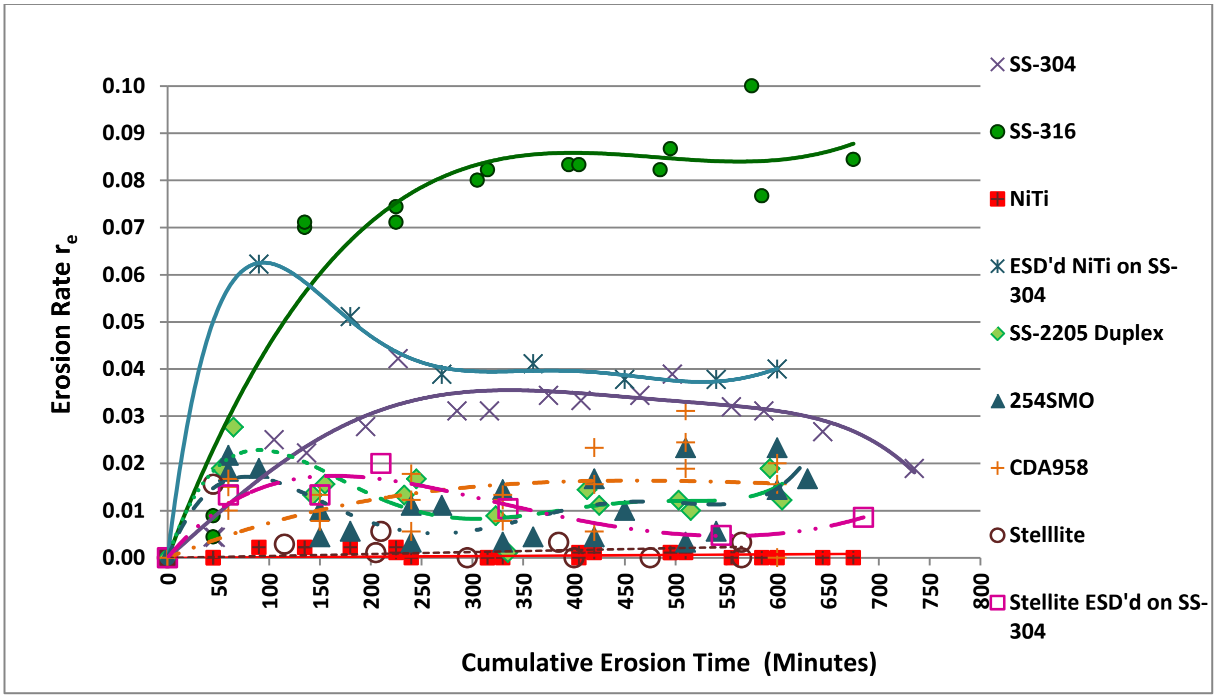

Figure 4-8 Erosion Rate Trend lines for Tested Alloys

The time-weighted and averaged erosion rates, ranked from lowest to highest over all of the cycles except the first, are given in Table 4-2.

Table 4-2 Averaged Erosion Rates for CE Tested Alloys

Material	r_e (mg/min) (wt. avg.)
Nitinol, pure	0.0008
Stellite 6®	0.0018
254SMO	0.0103
Stellite 6®, ESD	0.0114
SS-2205	0.0123
CDA958	0.0140
SS-304	0.0293
Nitinol, ESD	0.0411
SS-316L	0.0805

From both the graph and the data, some characteristics are immediately apparent:

- Nitinol and Stellite alloys (non-ESD) have the lowest CE rates.
- SS-316L suffers the greatest erosion rate, second to ESD Nitinol on SS-304, followed by SS-304 itself.
- The erosion rates for ESD'd Stellite, 254SMO, CDA958, and SS-2205 appear to span similar ranges throughout the tested period, and are in between the rates for the least- and most-susceptible materials.

Thus, electro-spark-deposited Nitinol on an SS-304 substrate has a higher erosion rate than both pure Nitinol and even the substrate itself. The ESD process appears to produce a microstructure that makes the Nitinol deposit much more vulnerable to CE, two orders of magnitude greater than pure Nitinol.

4.3.2 Comparison of Erosion-Resistance Values

The erosion resistance for a material at each recorded cumulative CE time was calculated as:

$$\boldsymbol{R_e} = \frac{(\Delta \boldsymbol{t})_{\boldsymbol{cuml}}}{(\Delta \boldsymbol{m})_{\boldsymbol{cuml}}}$$

where $\boldsymbol{R_e}$ is the erosion resistance and the subscript *cuml* indicates that Δt and Δm represent the amounts of cumulative time and mass loss, respectively. Erosion rates were not precisely constant over time, so it would be expected that erosion resistance would change over time as well. Being a reciprocal of the small $\boldsymbol{r_e}$, however, renders it relatively steady-state on a graph after one or two exposure cycles. Figure 4-9 shows the erosion resistance for each alloy throughout the course of the CE testing, plotted on a logarithmic scale.

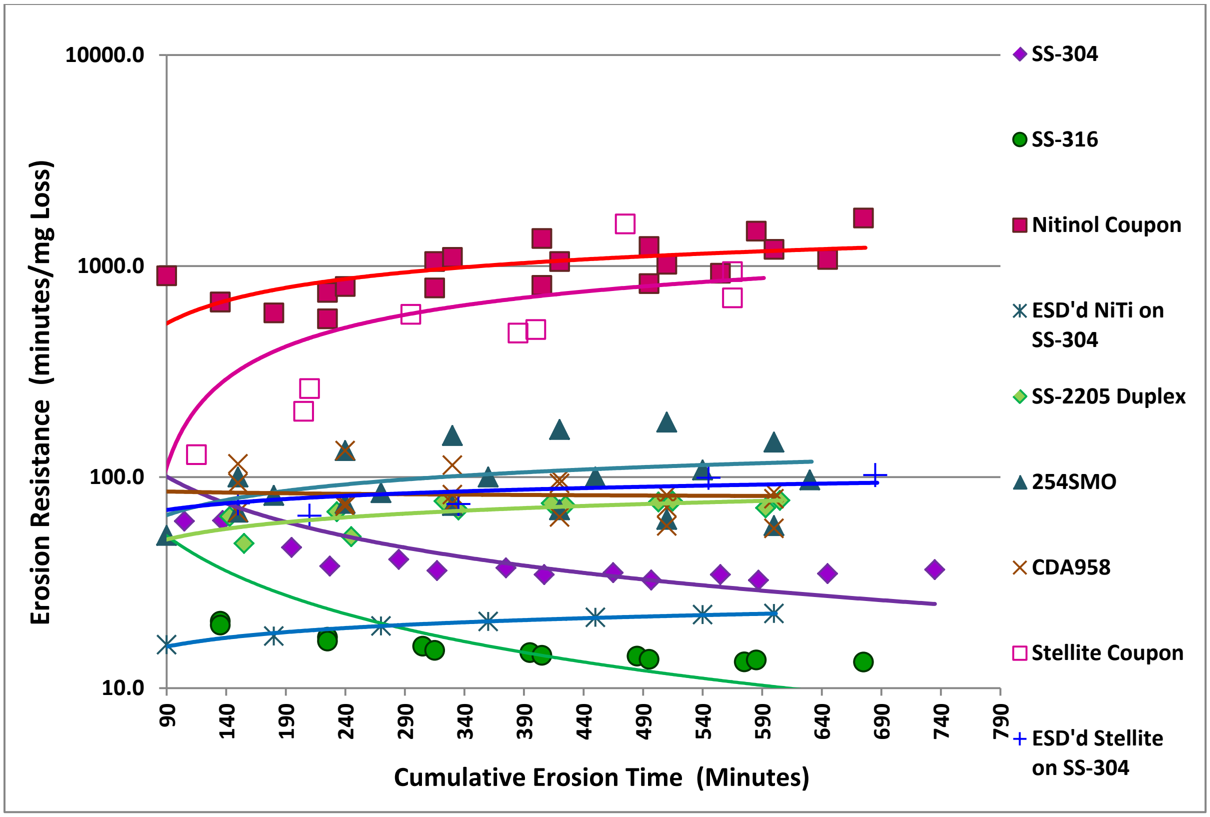

Figure 4-9 Erosion Resistance Trend Lines for Tested Alloys on a Log Scale

Table 4-3 lists the time weighted and averaged erosion resistance (R_e) values, over cycles starting from 90 minutes, from highest to lowest.

Table 4-3 Erosion Resistance R_e of the Tested Alloys

Material	R_e (mg/min)
Nitinol, pure	1200
Stellite 6®, pure	700
254SMO	102
Stellite 6®, ESD	90
SS-2205	74
CDA958	73
SS-304	34
Nitinol, ESD	23
SS-316L	13

ESD Nitinol is almost two orders of magnitude less resistant than Nitinol alloy. ESD'd Stellite, 254SMO and CDA958 are more susceptible to CE than pure Nitinol, but still erode less than ESD Nitinol. SS-316L is the least resistant of all the materials tested.

4.3.3 Correlation to Other Material Properties

Table 4-4 presents the values for tensile strength for each material (non-ESD) taken from their respective mill certifications, ranked from highest to lowest, along with the $\boldsymbol{r_e}$ values obtained by EXWC's testing.

Table 4-4 Comparison of Tensile Strength to Erosion Rate r_e

Material	**Tensile Strength (ksi)**	$\boldsymbol{r_e}$ (mg/min)
Nitinol, Coupon/Wire	195 [9] [10]	0.0008
Stellite®, Coupon	145 [17]	0.0018
SS-2205	124 [13]	0.0123
254SMO	108 [14]	0.0103
CDA958	96 [15]	0.0140
SS-304	90 [11]	0.0293
SS-316L	88 [12]	0.0805

Tensile strength order correlates to the order of erosion resistance from highest to lowest, with the exception of 254SMO, which is nonetheless very close to SS-2205. Because of the small number of samples tested in this study, and the variances found between duplicate coupons of 254SMO, the correlation is imperfect.

Cavitation erosion requires large external forces to break the bonds between particles in an alloy and tensile strength is a measure of how much pressure, or force per area, is required to induce a material to fracture. Therefore, we may expect that a material's erosion resistance is well-correlated with tensile strength, although this is dependent on the type of load applied. Generally, within groups of similar materials, the cavitation erosion resistance increases with increasing hardness and tensile strength. [25]

Figure 4-10 is a graph of reported tensile strengths vs. the tested erosion rates, averaged for each non-ESD material and fit with a power regression trend line.

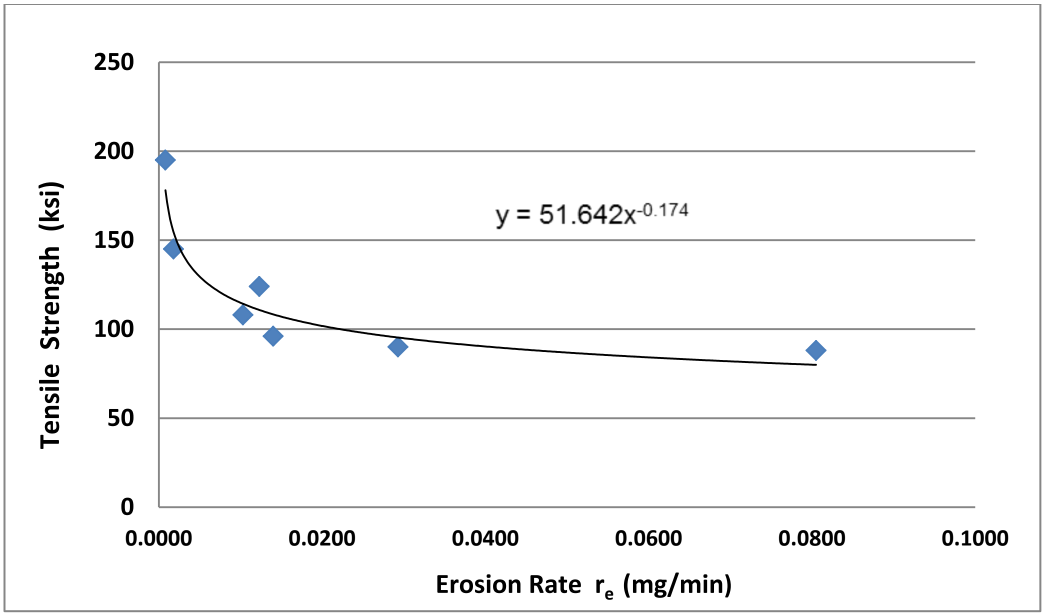

Figure 4-10 Alloy Tensile Strength vs. Erosion Rate, r_e

The trend for tensile strength vs. r_e indicates there is also a relationship between R_e and tensile strength. If the value of the tensile strength is known, then r_e can be predicted using the power-fit curve above, and R_e can in turn be predicted through its inverse relationship with r_e.

An approximate relationship between the hardness and the tensile strength (of steel) is:

- Tensile Strength (psi) ~ 515 x HB Hardness when HB $\leq$ 175
- Tensile Strength (psi) ~ 490 x HB Hardness when HB > 175

where *HB* is the Brinnell Hardness of the material, as measured with a standard indenter and a 3000 kgf (kilogram force) load. [26] Within a group of similar metals: [25]

$$\text{Erosion Resistance } \mathbf{R_e} \sim 1/(\text{Tensile Strength})^2 \text{ or } 1/(\text{Hardness})^2$$

Not all physical and mechanical properties of alloys correlate with erosion resistance. One example is Young's modulus. Young's modulus, "*E*", is a measure of stiffness, not strength. It is calculated for metals by dividing the tensile stress by the extensional strain in the initial (elastic) linear portion of the stress–strain curve. Below are the values of Young's modulus for each alloy (non-ESD) per literature values, listed from highest to lowest.

- Stellite 6® 210 GPa [17]
- SS-2205: 200 GPa [27]
- 254SMO: 195 GPa [28]

- SS-304: 193 GPa [29]
- SS-316L: 193 GPa [30]
- CDA958: 114 GPa [31]
- NiTi (austenite): 83 GPa [32]
- NiTi (martensite): 28-41 GPa [32]

Notice that Nitinol and Stellite are on opposite ends of the scale, even though both materials have superior cavitation erosion resistance. Additionally, while SS-316L was the alloy most prone to erosion during CE testing, its Young's modulus is quite similar to moduli of SS-2205, 254SMO, and SS-304. These materials' erosion rates and resistance values diverge significantly. Young's modulus does not correlate to the erosion resistance of the tested materials.

4.4 Microscopy

Subsequent to the cavitation erosion testing, microscopy was used to examine the intact and eroded areas of the ESD materials and SS-304 substrate. Figures 4-11 through 4-18 show images capturing the microscopic appearance of the ESD'd Nitinol and Stellite, both intact and eroded areas, on the SS-304 substrate. Figures 4-19 and 4-20 show the stainless substrate itself, intact and eroded, respectively. Figures 4-21 and 22 reveal the 3D color mapping of both materials.

The cavitation of ESD Nitinol on the SS-304 substrate is clearly visible to the naked eye as seen in Figure 4-13. The small amount of erosion on the ESD Stellite is much less visible (Figure 4-14). At 100X magnification (Figures 4-15 and 4-16), one can see that both ESD Nitinol and Stellite have rather rough surfaces. Stellite is particularly so. It is generally accepted that a smoother surface is one parameter that contributes to increased cavitation resistance for metals. However, in spite of its roughness, the ESD Stellite had significantly improved cavitation erosion resistance compared to the Series 300 stainless, and the ESD Nitinol.

ESD operating parameters may be adjusted to achieve a finer ESD surface, e.g. using a higher electrode rotation speed, using a hollow electrode, or increasing the sparking frequency. Alternately, a finishing step could be implemented to smooth the surface after ESDing. However, for hard overlays such as Nitinol and Stellite, diamond or electron-beam discharge machining would be required to "knock down" the surface roughness. [33] It would be difficult to achieve an even surface in the field.

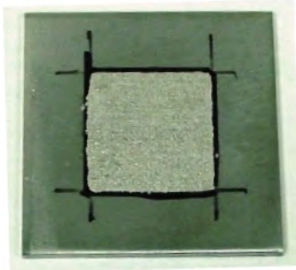

Figure 4-11 Nitinol ESD'd on SS-304

Figure 4-12 Stellite ESD'd on SS-304

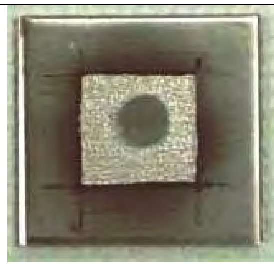

Figure 4-13 Cavitation Eroded ESD Nitinol

Figure 4-14 Cavitation Eroded ESD Stellite (slightly silvered area)

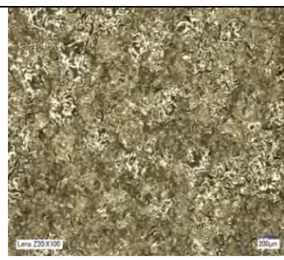

Figure 4-15 100X Close Up of Intact ESD Nitinol

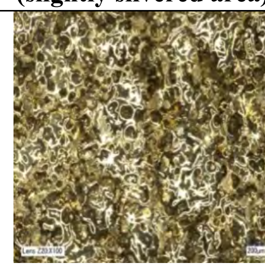

Figure 4-16 100X Close Up of Intact ESD Stellite

Figure 4-17 100X Edge of Fully Eroded Area of ESD Nitinol

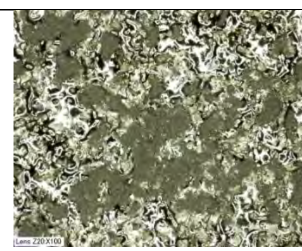

Figure 4-18 100X Slightly Eroded Area of ESD Stellite

Figure 4-19 100X Intact SS-304 Coupon Surface

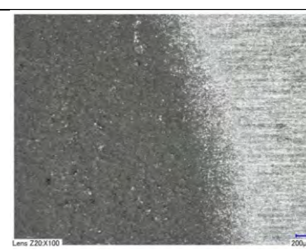

Figure 4-20 100X Eroded Edge of SS-304 Coupon

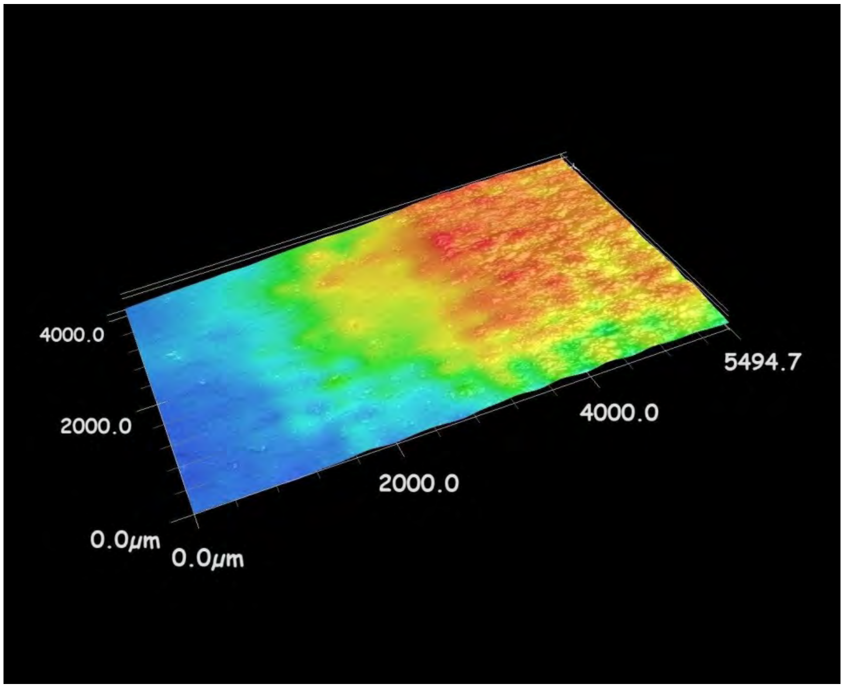

Figure 4-21 3D 50X Image of Eroded (Blue-Green Areas) vs Intact (Red-Orange Areas) ESD'd Nitinol on SS-304

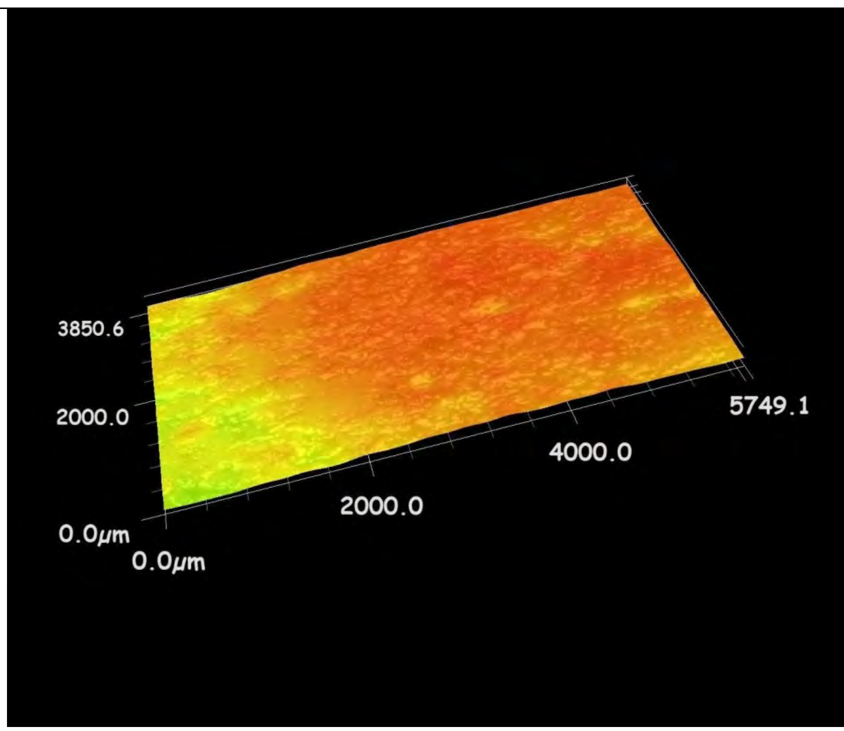

Figure 4-22 3D 50X Image of Partially Eroded (Yellow-Green Areas) vs Intact (Red-Orange Areas) ESD'd Stellite on SS-304

5.0 CONCLUSION

This investigation examined the feasibility of merging electro-spark deposition with erosion resistant, superelastic Nitinol to provide a viable product for improving cavitation erosion resistance. However, the laboratory evaluation revealed that ESD Nitinol is far more vulnerable to CE than pure Nitinol, and is even more vulnerable than SS-304. The tested ESD Nitinol was about two orders of magnitude less resistant than pure Nitinol, suggesting that ESD deposited Nitinol on less resistant impeller materials would not adequately withstand CE in seawater-machinery applications. The alloys 254SMO, SS-2205, and CDA958 would produce significantly improved impeller and similar components over those made from 300 series stainless steel.

ESD is capable of producing high-erosion-resistance layers with other materials, as demonstrated with Stellite 6®. Equipment is available commercially to operate remotely in the field. The main disadvantage for ESD is its inability to deposit over large areas quickly. In order to coat a large component such as a ship propeller, an automated factory set-up would be required and would take many hours to complete. Field ESD is best suited for hand-held sized coating and overlay products. The necessity for operator proficiency is also an issue.

EXWC has procured three identical centrifugal pumps in order to conduct a demonstration of ESD Stellite vs Nitinol at its Seawater Corrosion Laboratory. One pump impeller (SS-316) will receive Stellite deposit, another the Nitinol, and a third pump will contain an uncoated SS-316 impeller for control. Cavitation erosion will be induced in all three pumps either by holding the net-positive suction head available (NPSHA) steady while throttling the pump flow at the discharge head, or by holding the capacity constant and reducing the NPSHA by throttling the vacuum on the pump suction. The pumps will periodically be disassembled and the impellers inspected for damage. Additionally, bronze and cast iron impellers may be likewise tested. We anticipate that ESD Stellite will have superior performance in comparison to ESD Nitinol.

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APPENDIX A

CAVITATION EROSION TESTING RAW DATA

Table A 1 SS-304 Stainless Steel Coupon B

Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (02/22/13 and 02/26/13)
0	63.9334	0.0	0.0000		22	19.1	41	13	20	50	44	1.05	Sample: SS-304-B, washed with detergent and acetone then dried and weighed. New Tip = 2.7055-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
47	63.9332	0.2	0.0043	235.0	23	19	42	15	20	50	44	1.05	Tip face still shiny, slight outer wear ring present. Coupon spot just visible.
48					23	19	41		20	50	44	1.05	
137	63.9312	2.2	0.0222	62.3	23	19	41	14	20	50	44	1.05	Tip face somewhat dulled and outer wear ring more prominent. Coupon spot very visible.
138					23	19.1	40		20	50	44	1.05	
227	63.9274	6.0	0.0422	37.8	23	19	40	15	20	50	44	1.05	Tip face inside is light gray and dull. Promnent outer wear ring. Coupon spot gray and prominent.
228					20	19.1	40		20	50	44	1.05	Started 02/26/13 with fresh water.
317	63.9246	8.8	0.0311	36.0	24	19	42	14	20	50	44	1.05	Tip face now uniformly gray and dull. Coupon spot dark gray.
318					23	19	40		20	50	44	1.05	
407	63.9216	11.8	0.0333	34.5	23	19	40	14	20	50	44	1.05	Tip face uniformly gray. Coupon spot dark gray.
408					22	19.1	40		20	50	44	1.05	
497	63.9181	15.3	0.0389	32.5	23	19	39	14	20	50	44	1.05	Tip face uniformly dark gray. Coupon spot dark gray.
498					23	19.1	39		20	50	44	1.05	
587	63.9153	18.1	0.0311	32.4	24	19	39	15	20	50	44	1.05	Tip face dark gray. Tip = 2.6920-gm. Coupon spot dark gray. Tip Wt difference = 0.0135-gm.

Table A 2 SS-304 Stainless Steel Coupon C

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (03/12/13, 03/13/13, and 03/14/13)
0	0	64.0931	0.0	0.0000		21	19	43	12	20	50	44	1.05	Sample: SS-304-C, washed with detergent and acetone then dried and weighed. New Tip = 2.7206-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
45	45	64.0929	0.2	0.0044	225.0	23	19	47	15	20	50	44	1.05	Tip face has out ring of wear, but otherwise still shiny. Coupon spot visible.
1	46					23	19.1	42		20	50	44	1.05	
60	105	64.0914	1.7	0.0250	61.8	23	19	42	13	20	50	44	1.05	Outer wear ring on tip face now wider (middle of face still somewhat shiny). Coupon spot more prominent.
1	106					21	19	42		20	50	44	1.05	03/13/13 Re-Started with fresh water.
90	195	64.0889	4.2	0.0278	46.4	23	19	43	15	20	50	44	1.05	Tip face now fairly gray. Coupon spot now almost uniformly gray.
1	196					22	19	44		20	50	44	1.05	
90	285	64.0861	7.0	0.0311	40.7	23	19	44	14	20	50	44	1.05	Tip face and Coupon spot now uniformly gray. Water getting gray.
1	286					23	19.1	44		20	50	44	1.05	
90	375	64.0830	10.1	0.0344	37.1	23	19	44	14	20	50	44	1.05	Tip face and Coupon spot uniformly gray.
1	376					23	19	42		20	50	44	1.05	
90	465	64.0799	13.2	0.0344	35.2	23	19	43	14	20	50	44	1.05	Tip face and Coupon spot uniformly gray.
1	466					22	19.2	40		20	50	44	1.05	03/14/13 Re-Started with same water. Did not move the beaker.
90	555	64.0770	16.1	0.0322	34.5	23	19	41	14	20	50	44	1.05	Tip face and Coupon spot uniformly darker gray. Water very gray now.
1	556					23	19	39		20	50	44	1.05	
90	**645**	64.0746	18.5	0.0267	34.9	24	19	38	14	20	50	44	1.05	Tip face and Coupon spot very uniformly gray. Used Tip = 2.6986-gm. Tip Wt difference = 0.0220-gm.
90	735	64.0729	20.2	0.0189	36.4	23	19	41	14	20	50	44	1.05	Ran on 03/19/13 with new Tip to see what would happen. Weight loss rate dropped, possibly because period of steady weight loss is past, but possibly because probe tip and spot not aligned as they were previously?

Table A 3 SS-316 Stainless Steel Coupon A

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (02/20/13, 02/21/13, & 02/22/13)
0	0	63.0071	0.0	0.0000		21	19	41	14	20	50	44	1.05	Sample: SS-316-A, washed with detergent and acetone then dried and weighed. New Tip = 2.7332-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
45	45	63.0069	0.2	0.0044	225.0	22	19	41	14	20	50	44	1.05	Tip face has outer wear ring. Coupon spot visible.
90	135	63.0006	6.5	0.0700	20.8	23	19	41	15	20	50	44	1.05	More wear in inner tip face now, and dull. Coupon spot gray and very prominent.
1	136					22	19.1	42		20	50	44	1.05	
90	225	62.9942	12.9	0.0711	17.4	23	19	44	15	20	50	44	1.05	Tip face becoming more uniformly gray. Coupon spot now very prominent.
1	226					23	19	43		20	50	44	1.05	
90	305	62.9878	19.3	0.0800	15.8	23	19	45	16	20	50	44	1.05	Tip face now uniformly gray. Coupon spot dark gray and very prominent.
1	306					23	19.1	42		20	50	44	1.05	
90	395	62.9803	26.8	0.0833	14.7	23	19	42	15	20	50	44	1.05	Tip face now darker gray, uniform wear. Coupon spot is darker. Water getting "gray".
1	396					23	19.1	41		20	50	44	1.05	
90	485	62.9729	34.2	0.0822	14.2	23	19	41	15	20	50	44	1.05	Tip face dark gray and uniformly worn. Coupon spot dark.
1	486					21	19.1	42		20	50	44	1.05	02/22/13 - Fresh water.
90	575	62.9639	43.2	0.1000	13.3	23	19	42	16	20	50	44	1.05	Tip face very uniformly gray. Coupon spot dark gray. Tip = 2.7232-gm. Tip Wt difference = 0.0100-gm.

Table A 4 SS-316 Stainless Steel Coupon B

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (03/14/13, 03/15/13, and 03/18/13)
0	0	63.1586	0.0	0.0000		22	19.1	40	12	20	50	44	1.05	Sample: SS-316-B, washed with detergent and acetone then dried and weighed. New Tip = 2.6982-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
45	45	63.1582	0.4	0.0089	112.5	23	19	40	14	20	50	44	1.05	Tip face has very slight outer ring of wear. Coupon spot very visible.
90	135	63.1518	6.8	0.0711	19.9	23	19	39	14	20	50	44	1.05	Tip face outer wear ring more prominent. Coupon spot mostly gray now.
1	136					21	19.1	39		20	50	44	1.05	03/15/13 Restart
90	225	63.1451	13.5	0.0744	16.7	23	19	40	14	20	50	44	1.05	Tip face outer wear ring prominent. Coupon spot uniformly gray.
1	226					22	19.1	40		20	50	44	1.05	
90	315	63.1377	20.9	0.0822	15.1	23	19	40	14	20	50	44	1.05	Tip face now starting to wear in middle. Coupon spot uniformly darker gray.
1	316					23	19.1	39		20	50	44	1.05	
90	405	63.1302	28.4	0.0833	14.3	23	19	40	13	20	50	44	1.05	Tip face becoming gray. Coupon spot uniformly darker gray.
1	406					22	19.1	39		20	50	44	1.05	
90	495	63.1224	36.2	0.0867	13.7	23	19	38	13	20	50	44	1.05	Tip face now mostly uniformly gray. Coupons spot uniformly darker gray.
1	496					21	19	37		20	50	44	1.05	03/18/13 Restart with same water. Beaker unmoved.
90	585	63.1155	43.1	0.0767	13.6	23	19	38	11	20	50	44	1.05	Tip face uniformly gray. Coupon spot prominent and uniformly gray.
1	586					22	19	38		20	50	44	1.05	
90	675	63.1079	50.7	0.0844	13.3	23	19	38	14	20	50	44	1.05	Tip face uniformly gray. Coupon spot prominent and uniformly gray. Tip = 2.6876-gm. Tip Wt difference = 0.0106-gm.

Table A 5 SS-2205 Duplex Stainless Steel Coupon 01

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (04/23/14, 04/24/14, and 04/25/14)
0	0	59.0065	0.0	0.0000		21	19	48		20	50	44	1.05	Sample: SS-2205-01 Duplex, washed with detergent then dried and weighed. New Tip (Not Weighed). TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
53	53	59.0055	1.0	0.0189	53.0	23	19	48		20	50	44	1.05	Tip face has very slight outer ring of wear. Coupon spot visible. Weight loss could be from sonication-cleaning. Test another coupon that as been prevously cleaned in sonication bath.
90	143	59.0043	2.2	0.0133	65.0	23	19	47		20	50	44	1.05	
90	233	59.0031	3.4	0.0133	68.5	23	19	43		20	50	44	1.05	Water slightly gray. Ended test for the day.
90	323	59.0023	4.2	0.0089	76.9	23	19	40		20	50	44	1.05	04/24/14. Fresh water. Spot prominent.
90	413	59.0010	5.5	0.0144	75.1	22	19	38		20	50	44	1.05	Water quite gray. Coupon spot more gray.
										20	50	44	1.05	
90	503	58.9999	6.6	0.0122	76.2	22	19	41		20	50	44	1.05	Water quite gray. Ended test for the day.
										20	50	44	1.05	
90	593	58.9982	8.3	0.0189	71.4	21	19	40		20	50	44	1.05	04/25/14. Fresh water. Spot prominent. Ti Tip now uniformly gray. End Test.

Table A 6 SS-2205 Duplex Stainless Steel Coupon 02

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (04/25/14, 04/28/14)
0	0	58.1248	0.0	0.0000		22	19	40		20	50	44	1.05	Sample: SS-2205-02 Duplex, sonicated in bath with detergent then dried and weighed. New Tip (Not Weighed). TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
65	65	58.1230	1.8	0.0277	36.1	23	19	41		20	50	44	1.05	Lost a bit more weight than Sample 2205-01 (which was not sonication cleaned before use), so weight loss of 2205 is not from being "cleaned" by the device in the initial 60-minutes.
90	155	58.1216	3.2	0.0156	48.4	22	19	40		20	50	44	1.05	Spot very prominent. We are trying to always orient the sample, tray , and sonicator shaft the same from session to session to ensure cavitation erosion is occuring in the same area each time.
90	245	58.1201	4.7	0.0167	52.1	23	19	41		20	50	44	1.05	End test for weekend.
90	335	58.1200	4.8	0.0011	69.8	22	19	39		20	50	44	1.05	Resume testing 04/28/14. Weight aboration occurred. Sample before test APPEARED to weigh 58.1231-gm, but then scale read 0.0042-gm after Miguel took coupon off.
90	425	58.1190	5.8	0.0111	73.3	22	19	38		20	50	44	1.05	
										20	50	44	1.05	
90	515	58.1181	6.7	0.0100	76.9	22	19	37		20	50	44	1.05	
										20	50	44	1.05	
90	605	58.1170	7.8	0.0122	77.6	22	19	38		20	50	44	1.05	

Table A 7 254SMO Austenitic 6% Mo Alloy Coupon 01

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	61.0687	0.0	0.0000		22	19	40		20	50	44	1.05	No change of water from 0 to 540 min
90	90	61.0670	1.7	0.0189	52.9	22	19	41		20	50	44	1.05	
90	180	61.0665	2.2	0.0056	81.8	22	19	40		20	50	44	1.05	
90	270	61.0655	3.2	0.0111	84.4	22	19	39		20	50	44	1.05	
90	360	61.0651	3.6	0.0044	100.0	22	19	38		20	50	44	1.05	
90	450	61.0642	4.5	0.0100	100.0	22	19	37		20	50	44	1.05	
90	540	61.0637	5.0	0.0056	108.0	22	19.1	37		20	50	44	1.05	
90	630	61.0622	6.5	0.0167	96.9					20	50	44	1.05	

Table A 8 254SMO Austenitic 6% Mo Alloy Coupon 02

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	60.5838	0.0	0.0000		22	19	38		20	50	44	1.05	Beaker water replaced with new deionized water after 240 min and after 420 min
60	60	60.5827	1.1	0.0183	54.5	23	19	39		20	50	44	1.05	
90	150	60.5823	1.5	0.0044	100.0	22	19	38		20	50	44	1.05	
90	240	60.5820	1.8	0.0033	133.3	23	19	40		20	50	44	1.05	
90	330	60.5817	2.1	0.0033	157.1	21	19	38		20	50	44	1.05	
90	420	60.5813	2.5	0.0044	168.0	22	19	43		20	50	44	1.05	
90	510	60.5810	2.8	0.0033	182.1	22	19	41		20	50	44	1.05	
90	600	60.5797	4.1	0.0144	146.3					20	50	44	1.05	

Table A 9 254SMO Austenitic 6% Mo Alloy Coupon 03

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	60.2284	0.0	0.0000		23	19	46		20	50	44	1.05	First 1.25 min: Power overload. Replaced tip, but not beaker water. Remaining 58.75 min of first test: Regular operation.
60	60	60.2271	1.3	0.0217	46.2	23	19	49		20	50	44	1.05	
90	150	60.2262	2.2	0.0100	68.2	23	19	49		20	50	44	1.05	
90	240	60.2252	3.2	0.0111	75.0	23	19	49		20	50	44	1.05	
90	330	60.2239	4.5	0.0144	73.3	23	19	49		20	50	44	1.05	
90	420	60.2224	6.0	0.0167	70.0	23	19	53		20	50	44	1.05	
90	510	60.2203	8.1	0.0233	63.0	23	19	60		20	50	44	1.05	
90	600	60.2182	10.2	0.0233	58.8					20	50	44	1.05	

Table A 10 CDA958 Nickel Aluminum Bronze Coupon 01

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	62.5494	0.0	0.0000		23	19	60		20	50	44	1.05	Beaker water replaced with new deionized water after 240 min and after 420 min. Temperature in most tests rose to 25 deg C 5 min after start.
60	60	62.5484	1.0	0.0167	60.0	21	19	60		20	50	44	1.05	
90	150	62.5472	2.2	0.0133	68.2	22	19	58		20	50	44	1.05	
90	240	62.5461	3.3	0.0122	72.7	23	19	59		20	50	44	1.05	
90	330	62.5454	4.0	0.0078	82.5	23	19	60		20	50	44	1.05	
90	420	62.5449	4.5	0.0056	93.3	22	19	57		20	50	44	1.05	
90	510	62.5432	6.2	0.0189	82.3	23	19	61		20	50	44	1.05	
90	600	62.5418	7.6	0.0156	78.9					20	50	44	1.05	

Table A 11 CDA958 Nickel Aluminum Bronze Coupon 02

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	62.5877	0.0	0.0000		23	19	40		20	50	44	1.05	Beaker water replaced with new deionized water after 240 min and after 420 min. Why did coupon erode much more than usual between 420 and 510 min, while virtually not at all between 510 and 600 min? Theresa suspects a problem with the tip.
60	60	62.5871	0.6	0.0100	100.0	22	19	39		20	50	44	1.05	
90	150	62.5864	1.3	0.0078	115.4	21	19	39		20	50	44	1.05	
90	240	62.5859	1.8	0.0056	133.3	23	19	40		20	50	44	1.05	
90	330	62.5848	2.9	0.0122	113.8	23	19	41		20	50	44	1.05	
90	420	62.5834	4.3	0.0156	97.7	23	19	40		20	50	44	1.05	
90	510	62.5806	7.1	0.0311	71.8	23	19	41		20	50	44	1.05	
90	600	62.5806	7.1	0.0000	84.5					20	50	44	1.05	

Table A 12 CDA958 Nickel Aluminum Bronze Coupon 03

Test Time (minutes)	Cumulative Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Std Freq & Ampl (Watts)	Sonicator Power at 100% Ampl in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Ampl % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES
0	0	61.9386	0.0	0.0000		23	19.1	39		20	50	44	1.05	Beaker water replaced with new deionized water after 240 min and after 420 min. All temperature and power measurements taken at 1 min after start of each cycle. One week passed between end of 240 cumulative minutes and beginning of next cycle.
60	60	61.9377	0.9	0.0150	66.7	22	19.1	41		20	50	44	1.05	
90	150	61.9370	1.6	0.0078	93.7	23	19.1	41		20	50	44	1.05	
90	240	61.9354	3.2	0.0178	75.0	22	19.1	42		20	50	44	1.05	
90	330	61.9342	4.4	0.0133	75.0	23	19.1	41		20	50	44	1.05	
90	420	61.9321	6.5	0.0233	64.6	24	19	45		20	50	44	1.05	
90	510	61.9299	8.7	0.0244	58.6	23	19.1	43		20	50	44	1.05	
90	600	61.9281	10.5	0.0200	57.1					20	50	44	1.05	

Table A 13 Nitinol Coupon A1

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (02/15/13 and 02/19/13)
0	0	25.0390	0.0	0.0000		20	19	44	17	20	50	44	1.05	Sample: 2"x2"x0.059" NiTi-A1, a NiTi coupon from Johnson-Matthey. New Tip = 2.7326-gm. (In-air watts at 100% Ampl = 17!) TRAY used throughout test. (Because Coupon turning in beaker - not still - had to improvize a "holder" - a shallow plastic tray that just fits the inside diameter of the beaker).
5	5					22	19	44		20	50	44	1.05	
13	13					23	19	45		20	50	44	1.05	
30	30					23	19	45		20	50	44	1.05	
45	45	25.0390	0.0	0.0000		23	19	46	24	20	50	44	1.05	Tip ever so slightly eroded - barely visible, and face still shiny. Coupon shows a barely visible "shadow". Initial sonication appears to "clean" the coupon, even if it has been cleaned manually before, so use the 1st sonication
1	46					21	19	45		20	50	44	1.05	
3	48					23	19.2	45		20	50	44	1.05	
39	84					23	19	46		20	50	44	1.05	
45	90	25.0389	0.1	0.0022	900.0	23	19	46	25	20	50	44	1.05	Tip is same as before but now with definite outer ring pattern on face. The coupon has "shadow" of a spot forming.
1	91					22	19	44		20	50	44	1.05	
90	180	25.0387	0.3	0.0022	600.0	23	19	46	25	20	50	44	1.05	Tip face is now slightly duller. Coupon's "shadow" spot is slightly more visible.
1	181					22	19	43		20	50	44	1.05	
60	240	25.0387	0.3	0.0000	800.0	23	19	44	23	20	50	44	1.05	Tip face starting to look gray. Coupon's "shadow" spot still slightly more visible. Water still looks clean.
11	251					23	19	47		20	50	44	1.05	Started at 09:40 on 02/19/13 with fresh water. Weight recorded after being in dessicator over the weekend + brief heating in oven, then cooling.
86	326					23	19	46		20	50	44	1.05	
90	330	25.0387	0.3	0.0000	1100.0	23	19	46	22	20	50	44	1.05	Tip face looking grayer. Coupon shadow visible.
60	390					23	19	45		20	50	44	1.05	
90	420	25.0386	0.4	0.0011	1050.0	23	19	45	24	20	50	44	1.05	Tip face now uniform gray. Coupon shadow spot about the same.
90	510	25.0385	0.5	0.0011	1020.0	23	19	44	26	20	50	44	1.05	Tip face now uniform gray. Coupon shadow spot about the same.
90	600	25.0385	0.5	0.0000	1200.0	23	19	45	26	20	50	44	1.05	Tip face very uniform gray. Coupon shadow spot about the same. Used Tip = 2.7270-gm (cleaned and dried). Water still looks clean. Tip Wt difference = 0.0056-gm.

Table A 14 Nitinol Coupon B1

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (03/18/13, 03/19/13, and 03/20/13)
0	0	25.2435	0.0	0.0000		22	19	42	14	20	50	44	1.05	Sample: 2"x2"x0.059" NiTi-B1, a NiTi coupon from Johnson-Matthey. Washed with detergent and acetone then dried and weighed. New Tip = 2.7216-gm.
45	45	25.2435	0.0	0.0000		23	19	43	13	20	50	44	1.05	Tip face has outer ring of wear, else still shiny. Barely visible "shadow" of a spot on Coupon.
										20	50	44	1.05	
90	135	25.2433	0.2	0.0022	675.0	23	19	42	14	20	50	44	1.05	Tip face outer wear ring prominent, and some wear inside now. Coupon "shadow" spot slightly more visible.
1	136					22	19.1	43		20	50	44	1.05	03/19/13 Restarted with fresh water.
90	225	25.2432	0.3	0.0011	750.0	23	19	48	14	20	50	44	1.05	Tip face now appears uniformly worn. Coupon shadow visible.
1	226					22	19.1	43		20	50	44	1.05	
90	315	25.2432	0.3	0.0000	1050.0	23	19	43	14	20	50	44	1.05	Tip face now appears uniformly worn. Coupon shadow visible.
1	316					22	19.1	41		20	50	44	1.05	
90	405	25.2432	0.3	0.0000	1350.0	23	19	42	14	20	50	44	1.05	Tip face uniformly worn gray. Coupon shadow spot easily visible.
1	406					21	19.1	40		20	50	44	1.05	03/20/13 Restarted with same water. Beaker unmoved. Water a bit gray.
90	495	25.2431	0.4	0.0011	1237.5	23	19	40	13	20	50	44	1.05	Tip face uniformly worn gray. Coupon shadow spot easily visible.
1	496					22	19.1	40		20	50	44	1.05	
90	585	25.2431	0.4	0.0000	1462.5	23	19	40	14	20	50	44	1.05	Tip face uniformly darker gray. Coupon shadow spot very visible. Water getting more gray.
1	586					23	19.1	39		20	50	44	1.05	
90	675	25.2431	0.4	0.0000	1687.5	23	19	43	13	20	50	44	1.05	Tip face uniformly darker gray. Coupon shadow spot very visible. Used Tip = 2.7018-gm. Tip Wt Diff = 0.0198-gm. Water is gray - from Tip wear, which is significantly more (by weight) than NiTi Coupon A1 Tip.

Table A 15 Nitinol Coupon B4

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (03/26/13, 03/27/13)
0	0	25.0455	0.0	0.0000		5	4.9	41	10	20	50	44	1.05	Sample: 2"x2"x0.059" NiTi-B4, a NiTi coupon from Johnson-Matthey. Washed with detergent and acetone then dried and weighed. New Tip = 2.6328-gm.
5	5					4	5.2	39		20	50	44	1.05	Ice in the water melts after about 3 minutes. Continuously adding ice results in the probe becoming too submerged in water (NOT good per the mfg instructions).
45	45	25.0455	0.0	0.0000		7	4.8	43	15	20	50	44	1.05	Tip face has very slight outer ring of wear, else still shiny. No shadow or spot of wear visible on Coupon.
5	50									20	50	44	1.05	
90	135	25.0453	0.2	0.0022	675.0	7	5	45	14	20	50	44	1.05	Tip face has definite outer ring plus wear pattern in middle. Coupon looks same - no visible shadow or spot.
5	140					6	5.2	47		20	50	44	1.05	
90	225	25.0451	0.4	0.0022	562.5	8	5	48	15	20	50	44	1.05	Tip face definitely worn now - not quite uniform across the face. Coupon "shadow spot" just now visible.
5	230					6	5.2	41		20	50	44	1.05	03/27/13 Restarted with fresh water.
90	315	25.0451	0.4	0.0000	787.5	8	5	37	16	20	50	44	1.05	Tip face mostly uniformly worn. Coupon shadow spot visible.
5	320					5	5	39		20	50	44	1.05	
90	405	25.0450	0.5	0.0011	810.0	8	5	40	16	20	50	44	1.05	Tip face uniformly worn. Coupon shadow spot plainly visible now.
5	410					6	5.1	40		20	50	44	1.05	
90	495	25.0449	0.6	0.0011	825.0	8	5	39	16	20	50	44	1.05	Tip face uniformly worn. Coupon shadow spot plainly visible now.
5	500					6	5.1	41		20	50	44	1.05	04/05/13 Restarted with fresh water.
60	555	25.0449	0.6	0.0000	925.0	7	5	42	17	20	50	44	1.05	Tip face uniformly worn. Coupon shadow spot plainly visible now.
5	560					6	5.1	41		20	50	44	1.05	
90	645	25.0449	0.6	0.0000	1075.0	7	5	39	17	20	50	44	1.05	Tip very uniformly gray. Coupon shadow spot very visible. Used Tip Wt. = 2.6246-gm.

Table A 16 ASAP ESD Nitinol on SS-304

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (02/27/13 and 02/28/13)
0	0	64.1344	0.0	0.0000		22	19	40	9	20	50	44	1.05	Sample: NiTi-ESD-SS304-1, washed with detergent and acetone then rinsed with dei-water, dried and weighed. New Tip = 2.6976-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
90	90	64.1288	5.6	0.0622	16.1	23	19	40	11	20	50	44	1.05	
1	91					24	19	43		20	50	44	1.05	Tip Face has outer wear ring, and inner wear ring. ESD NiTi exposed area has a definitive wear "spot".
90	180	64.1242	10.2	0.0511	17.6	24	19	43	13	20	50	44	1.05	Tip face now uniform light gray. Coupon spot is prominent!
1	181					22	19.1	44		20	50	44	1.05	
90	270	64.1207	13.7	0.0389	19.7	23	19	43	13	20	50	44	1.05	Tip face now uniformly medium gray. Coupon spot is prominent. Water now quite "gray".
1	271					22	19.1	41		20	50	44	1.05	Started 02/28/13 at ~08:30 with fresh water.
90	360	64.1170	17.4	0.0411	20.7	23	19	43	11	20	50	44	1.05	Tip face uniformly dark gray. Coupon spot increasingly prominent.
1	361					22	19.1	41		20	50	44	1.05	
90	450	64.1136	20.8	0.0378	21.6	23	19	42	12	20	50	44	1.05	Tip face uniformly dark gray. Coupon spot increasingly prominent.
1	451					22	19.1	41		20	50	44	1.05	
90	540	64.1102	24.2	0.0378	22.3	24	19	42	13	20	50	44	1.05	Tip face uniformly dark gray. Coupon spot very prominent.
1	541					23	19.1	41		20	50	44	1.05	
60	600	64.1078	26.6	0.0400	22.6	23	19	41	13	20	50	44	1.05	Tip face uniformly worn gray. Coupon spot very prominent. Definite ESD layer degradation. Tip = 2.6761-gm. Water quite gray again. Tip Wt difference = 0.0215-gm.

Table A 17 EXWC ESD Nitinol on SS-304

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (10/15/14 & 10/16/14)
0	0	63.9317	0.0	0.0000		21	19	48	0	20	50	44	1.05	Sample: EXWC NiTi-ESD-SS304, washed with detergent and acetone then rinsed with dei-water, dried and weighed. New Tip = 2.7215-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
85	85	63.9290	2.7	0.0318	31.5	23	19	50	1	20	50	44	1.05	
						21	19	47		20	50	44	1.05	Tip face has prominent rings of wear, not much shine left. Wear mark visible on NiTi surface. Water already slightly gray.
120	205	63.9256	6.1	0.0283	33.6	23	19	50	4	20	50	44	1.05	Tip face now uniformly gray. Eroded mark on NiTi more visible. Water quite gray.
						20	19	49		20	50	44	1.05	10/16/14 Fresh water.
180	385	63.9220	9.7	0.0200	39.7	24	19	51	6	20	50	44	1.05	Water gray. Tip face very uniformly dark gray. Coupon mark very prominent now.
						22	19	48		20	50	44	1.05	10/17/14 Same water.
140	525	63.9190	12.7	0.0214	41.3	23	19	50	7	20	50	44	1.05	Water quite gray now. Very gray wear spot on coupon, contrasting with surrounding semi-shiny ESD'd NiTi. Tip face very uniform dark gray.
						21	19	47	7	20	50	44	1.05	
150	675	63.9168	14.9	0.0147	45.3	24	19	48	7	20	50	44	1.05	Tip face very gray. Mark on coupon very prominent. NiTi may have been C.E.'d away after 1st session - the ESD'd NiTi on this coupon was quite thin, in comparison to the ASAP coupon. Water very gray.
										20	50	44	1.05	Weight loss line slope "sort of" follows that of regular SS-304 after the initial weight loss.
			63931.7	-94.6916	0.0					20	50	44	1.05	Eroded Ti Tip Weight = 2.6950-gm.

Table A 18 Stellite Coupon #1

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (09/10/14, 09/11/14, and 09/12/14)
0	0	110.5004	0.0	0.0000		22	19	36	1	20	50	44	1.05	Sample: Stellite-6 #1 Round Coupon, sonicated with detergent then rinsed with dei-water, dried and weighed. New Tip = 2.7218-gm. TRAY used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing. NO wear on coupon after 45-minutes.
45	45	110.4997	0.7	0.0156	64.3	24	19.1	36	0	20	50	44	1.05	
1	46					23	19	36		20	50	44	1.05	
70	115	110.4995	0.9	0.0029	127.8	23	19	36	0	20	50	44	1.05	NO wear on coupon. Tip has definite face wear, especially an outer ring. Water slightly gray.
1	116									20	50	44	1.05	
90	205	110.4994	1.0	0.0011	205.0	24	19.1	36	1	20	50	44	1.05	Tip face now evenly worn to a dark gray dull surface. Water more gray now.
1	206					22	19	36		20	50	44	1.05	
90	295	110.4999	0.5	-0.0056	590.0	24	19.1	36	1	20	50	44	1.05	No wear on coupon. Tip face uniformly dark gray.
1	296					22	19	36		20	50	44	1.05	
90	385	110.4996	0.8	0.0033	481.3	24	19	35	1	20	50	44	1.05	Tip face very dark gray and dull. Water gray.
1	386					22	19	38		20	50	44	1.05	Changed out the water 09/12/14.
90	475	110.5001	0.3	-0.0056	1583.3	24	19.1	37	2	20	50	44	1.05	Still NO easily visible wear on the coupon. (Possible faint stain when rotated under light.)
1	476					22	19	37		20	50	44	1.05	
90	565	110.4998	0.6	0.0033	941.7	23	19	37	0	20	50	44	1.05	NO wear on the coupon. (Possible faint stain when rotated under light.)
										20	50	44	1.05	Tip wt = 2.7037-gm. (Loss = 0.0181-gm)

Table A 19 Stellite Coupon #2

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES 10/07/14, 10/08/14)
0	0	110.8887	0.0	0.0000		21	19	42	2	20	50	44	1.05	Sample: Stellite-6 #2 Round Coupon, sonicated with detergent then rinsed with dei-water, dried and weighed. New Tip = 2.7031-gm. TRAY and elastomer spacers used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
68	68					22	19.1	49		20	50	44	1.05	
19	87					23	19	47		20	50	44	1.05	Let Sonicator probe run for 3-Hr and 30-min.
123	210	110.8879	0.8	0.0056	262.5	24	19	49	11	20	50	44	1.05	NO wear on coupon. Tip has definite face wear, especially an outer ring. Water slightly gray.
						21	19	48	11	20	50	44	1.05	Phenomenon of initial wt loss seen, as in previous testing, even when previously cleaned in sonicator.
190	400	110.8879	0.8	0.0000	500.0	24	19	46	7	20	50	44	1.05	10/08/14 - Used same water. Faint spot now visible on coupon. Tip face uniformly gray. Water dirty.
						22	19	47		20	50	44	1.05	
165	565	110.8879	0.8	0.0000	706.3	24	19	46		20	50	44	1.05	Faint mark on coupon. Tip face uniformly eroded and gray. Tip wt = 2.6876-gm. (Loss = 0.0155-gm)

Table A 20 EXWC ESD Stellite on SS-304

Test Time (minutes)	Cumulative Exposure Time (minutes)	Coupon Weight (gm)	Cumulative Weight Loss (mg)	Erosion Rate 1st Derivative Curve	Erosion Resistance Minutes/mg Curve	Water Temperature (C°)	Chiller Coil Temp (C°)	Sonicator Power in Water at Standard Frequency & Amplitude (Watts)	Sonicator Power at 100% Amplitude in Air (Watts)	Sonicator Frequency (K-Hz)	Amplitude (um)	Amplitude % of Probe Max (114-um)	Gap Distance between Tip and Coupon (mm)	NOTES (10/09/14)
0	0	63.9253	0.0	0.0000		21	19	45	0	20	50	44	1.05	Sample: Stellite-6 ESD'd onto SS304 Coupon. Coupon, sonicated with detergent then rinsed with dei-water, dried and weighed. New Tip = 2.7091-gm. TRAY and elastomer spacers used to hold coupon still. Coupon washed in dei-water and dried and cooled before each weighing.
60	60	63.9245	0.8	0.0133	75.0	23	19	43	0	20	50	44	1.05	
						21	19	42	0	20	50	44	1.05	Tip face slightly degraded, still shiny with outer ring wear. Accidentally did not place entire tip face over ESD'd area - bit of wear now on SS 304 bare surface. Otherwise hard to see any wear.
90	150	63.9233	2.0	0.0133	75.0	23	19	41	0	20	50	44	1.05	Flipped coupon around for remaining runs. Part of tip face is still over the original cavitated spot over ESD'd Stellite.
						22	19	40		20	50	44	1.05	Faint mark visible on coupon. Tip face starting to become gray with prominent outer wear ring.
60	210	63.9221	3.2	0.0200	65.6	23	19	41	0	20	50	44	1.05	Tip face becoming uniformly gray. Faint mark visible on coupon. Water somewhat gray.
						21	19	38		20	50	44	1.05	10/14/14 - Fresh water.
125	335	63.9208	4.5	0.0104	74.4	22	19	38	1	20	50	44	1.05	Water slightly gray. Tip face uniformly gray. Faint wear on coupon.
						21	19	38		20	50	44	1.05	
210	545	63.9198	5.5	0.0048	99.1	22	19	38	1	20	50	44	1.05	Water quite gray now. Tip face darker uniform gray. Faint wear on coupon looks the same as last check. Weight loss has slowed down per unit time. Stellite is probably excessively wearing the Ti tip.
						21	19	37		20	50	44	1.05	10/15/14 - Same Water.
140	685	63.9186	6.7	0.0086	102.2	22	19	37	2	20	50	44	1.05	Ti Tip Wt = 2.6794-gm. Faint but visible wear on the ESD'd Stellite coating now.

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