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The effect of fatigue on the corrosion resistance of common medical alloys

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Abstract

The effect of mechanical fatigue on the corrosion resistance of medical devices has been a concern for devices that experience significant fatigue during their lifespan and devices made from metallic alloys. The Food and Drug Administration had recommended in some instances for corrosion testing to be performed on post-fatigued devices [Non-clinical tests and recommended labeling for intravascular stents and associated delivery systems: guidance for industry and FDA staff. 2005: Food and Drug Administration, Center for Devices and Radiological Health], although the need for this has been debated [Nagaraja S, et al., *J Biomed Mater Res Part B: Appl Biomater* 2016, 8.] This study seeks to evaluate the effect of fatigue on the corrosion resistance of 5 different materials commonly used in medical devices: 316 LVM stainless steel, MP35N cobalt chromium, electropolished nitinol, mechanically polished nitinol, and black oxide nitinol. Prior to corrosion testing per ASTM F2129, wires of each alloy were split into subgroups and subjected to either nothing (that is, as received); high strain fatigue for less than 8 min; short-term phosphate buffered saline (PBS) soak for less than 8 min; low strain fatigue for 8 days; or long-term PBS soak for 8 days. Results from corrosion testing showed that the rest potential trended to an equilibrium potential with increasing time in PBS and that there was no statistical ($p > 0.05$) difference in breakdown potential between the fatigued and matching PBS soak groups for 9 out of 10 test conditions. Our results suggest that under these nonfretting conditions, corrosion susceptibility as measured by breakdown potential per ASTM F2129 was unaffected by the fatigue condition.

Keywords

corrosion; fatigue; cardiovascular

INTRODUCTION

The potential for implanted metallic medical devices to corrode and cause patient complications was recognized many years ago¹ and remains a concern today. Over the years, there have been a number of efforts to develop corrosion test standards which span general

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corrosion testing as well as medical device area specific testing (e.g. ASTM G31-12, ASTM F746-04, and ASTM F2129-08).² Multiple studies have been conducted to understand *in vivo* corrosion as well as to establish acceptance criteria for existing test methods.^{3,4} As became evident at the FDA Public Workshop – Cardiovascular Metallic Implants: Corrosion, Surface Characterization, and Nickel Leaching held on March 8 – 9, 2012 [7], there are still a number of outstanding questions for corrosion testing of medical devices. One of these questions is whether fatigue loading increases pitting corrosion susceptibility. The 2010 FDA Guidance for Industry and FDA Staff “Non-Clinical engineering testing and recommended labeling for intravascular stents and associated delivery systems”⁵ recommends that corrosion testing, specifically ASTM F2129, be performed on specimens that have been subjected to fatigue test conditions. This can present logistical, time, and sample size constraints for completion of a corrosion study. These challenges were highlighted at the 2012 FDA Corrosion Workshop and test results submitted by attendees did not show that fatigue loading prior to ASTM F2129 testing was more conservative than corrosion testing of as manufactured components.⁶ This work aims to test the hypothesis that fatigue testing reduces the pitting corrosion resistance via damage to the oxide layer.

Investigating the effect of strain and/or damage to alloys used in medical devices is not a new area of interest⁷ nor is the interplay between corrosion and fatigue crack growth.^{8–11} However, there has not been a systematic study on how fatigue testing itself affects the corrosion resistance of the fatigue sample. In this study, wires were selected as a generic test specimen that is expected to be representative of more complicated medical devices. While there are many methods of fatigue, rotary bend fatigue was selected for this work because it is a relatively common and well understood test method.^{12–14} Additionally, rotary bend wire fatigue allowed for convenient transferring of samples from the wire fatigue testers to the corrosion cell after fatigue testing. It should be noted that this work does not incorporate any fretting or overlapped fatigue conditions. This facilitated our ability to investigate the effect of fatigue induced oxide damage on the corrosion resistance of common medical alloys.

MATERIALS AND METHODS

Materials

For this study, five alloys were selected based on usage in medical devices: 316 LVM stainless steel (316 LVM), MP35N cobalt chromium (MP35N), electropolished nitinol (EP nitinol), mechanically polished nitinol (MP nitinol), and black oxide nitinol (BO nitinol). The 316 LVM stainless steel and MP35N cobalt chromium wires had a diameter of 0.178 mm while the nitinol (electropolished, mechanically polished, and black oxide) had a diameter of 0.500 mm. The different diameters were selected so that we could achieve the desired fatigue performance for each alloy. All wires were purchased from Fort Wayne Metals (Fort Wayne, IN). Wires were cleaned with acetone to remove any residual oils but were otherwise used as received.

Test Plan: Prior to corrosion testing, wire specimens of each alloy were split into groups and subjected to one of the following.

1. As received (i.e., no treatment)

2. High strain fatigue in phosphate buffered saline (PBS) for several minutes (i.e., to achieve 95% average fatigue life)
3. Short-term soak in PBS for several minutes.
4. Low strain fatigue in PBS for eight days (i.e., to 40 million cycles).

Long-term soak in PBS for 8 days.

The as received group serves as a baseline for comparing the effects of the varying fatigue and soak conditions. The high strain fatigue group was designed to be a “worst case” condition with fatigue cracks along the wire length prior to initiating corrosion testing. The low strain fatigue group was designed to be a more realistic scenario of a medical device which was subjected to a small alternating strain that was not expected to create surface cracks for a time that was equivalent to ~1 year of pulsatile loading (40 million cycles). Each of the fatigue groups had a matching control that was soaked in PBS without any fatigue loading so that we could more directly observe the effects of fatigue.

Fatigue testing

Fully reversed rotary beam fatigue testing was performed at 60 Hz using modified Valley Instruments (Positool Instruments, Brunswick, OH) wire fatigue testers as shown in Figure 1. To simulate physiological conditions and to minimize adiabatic effects, tests were conducted at 37°C in a PBS bath. To impose the desired strain, wires were routed through a 1.5 mm wide semicircular groove machined out of square Delrin blocks; this allowed for a uniform strain along the length of the wire. The amplitude strain, ϵ_a , in the wire is determined by:

$$\epsilon_a = \frac{d}{2R}$$

where d is the diameter of the wire and R is the radius of the mandrel. During testing, one end of the wire was clamped in a motor-driven chuck. The remaining length of the wire was inserted into the semi-circular groove, with the opposite end of the wire rotating freely outside the groove. The high and low strains for the 316 LVM and the MP35N wires were set to 0.50% and 0.14%, respectively. All three nitinol wires had the same high and low strain amplitudes, 1.00% and 0.40%, respectively.

The length of time for the low strain tests was selected so that the wires would undergo 40 million loading cycles (at an alternating strain value that was not expected to create surface cracks), which is approximately equal to one year of pulsatile loading (that is, as an artery contracts and dilates with changing blood pressure). The length of time for the high strain fatigue tests varied for each alloy. To determine the time, we initially conducted tests at the high strain level to determine the average number of cycles to failure ($n = 4$ for each alloy). We then took 95% of the average value and set it as the time for high strain fatigue testing. We selected the 95% value as a ‘worst case’ which was expected to create surface cracks in the oxide layer. By using different test times for the high strain fatigue condition for each alloy, we were able to ensure similar fatigue conditions across all five alloys. Any wire in the

high strain fatigue group that fractured prior to reaching 95% of its average fatigue life was discarded and another wire was fatigued in its place such that no fractured wires were ever subjected to corrosion testing. See Table I for fatigue parameters for all wires and conditions. Given the differing sizes of the grooved Delrin blocks used in the low and high strain fatigue testing, the low strain wires were longer than the high strain wires. The matching PBS soak wires were cut to be the same length as the fatigue specimens.

Corrosion testing

After being subjected to either fatigue testing or a PBS soak, wires were rinsed with deionized water, blotted dry, and then rinsed with acetone to remove any surface contaminants. As received wire specimens were subjected to an acetone rinse only. Wire was cut with wire cutters to a length of 65 mm so the same length of wire was tested for all conditions; a sample size of $n = 6$ was used for all conditions. For wires fatigued under the high strain conditions the 65 mm length was cut from the center of the fatigue specimen. For wires fatigued under the low strain conditions, three 65 mm lengths were cut from the 195 mm section at the apex of the fatigue specimen. This was the same for the respective PBS soak samples. These samples were then taped to a longer conductive rod and the approximately 15 mm overlap was painted with a conductive silver paint (Fast Drying Silver Paint, Ted Pella Inc., CA). Once dried, the cut end of the 65 mm length and the overlap region had three coats of insulated coating (Microstop, Tolber Chemical, AK) applied.

Corrosion testing was performed as described in ASTM F2129 “Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices.”⁴ A Model 263A (Princeton Applied Research, Oak Ridge, TN) or an Interface 1000 (Gamry, Warminster, PA) potentiostat was used to perform the corrosion testing; both experimental setups were in compliance with ASTM G5 “Standard Reference Test Method for Making Potentiostatic and Potentiodynamic Anodic Polarization Measurements.” A PBS bath was heated to 37°C in a water bath and was purged with compressed nitrogen (UN1066, Roberts Oxygen Co., MD) at 150 cc/min for 30 min. The sample was then inserted into the PBS bath and the open circuit potential was recorded for 60 min after which the potential was increased at a rate of 1 mV/s to 1000 mV and then reversed at a rate of 1 mV/s.

Following corrosion testing, samples were cleaned with deionized water and examined for evidence of pitting corrosion per ASTM F2129 using a digital microscope (KH-7700, Hirox, NJ). Additionally, some wire surfaces were examined with a scanning electron microscope (SEM, JEOL JSM-6390LV) at magnifications varying from 150x to 1000x. The SEM observations were conducted in both secondary and backscatter modes under high vacuum at 20 kV electron acceleration voltage. The rest potential (E_r) was defined as the potential at the end of the open circuit potential segment and the breakdown potential (E_b) was defined as the potential when a two decade increase in current occurred along with pitting confirmed by visual inspection.

Statistical Comparison

To assess the effect of fatigue on the corrosion behavior of the alloys, statistical tests were performed using MiniTab (Mintab Inc, State College, PA). Rest potential was assumed to have a normal distribution and we used two-sample *t*-tests with a Bonferroni Correction (α of $0.05/4 = 0.0125$) to compare between conditions. The Bonferroni Correction was employed in this way because we conducted four simultaneous statistical comparisons. With the exception of BO nitinol, all materials and conditions contained specimens that did not exhibit breakdown. In these cases breakdown potential, E_b , was considered to be 1000 mV and the data was censored. Therefore, log-rank analyses with a Bonferroni Correction (α of $0.05/4 = 0.0125$) were used to analyze breakdown potential and over potential for all materials except BO nitinol. None of the BO nitinol data sets were censored so two-sample *t*-tests with the Bonferroni Correction were used as described above to compare breakdown potential and over potential.

RESULTS

The fatigue to failure results that were used to calculate the 95% fatigue life threshold for the high strain condition in Table I can be seen in Table II. There is no fatigue to failure data for the low strain condition as that strain level was selected such that the wire would survive to 40 million cycles without initiation of surface cracks or fatigue failure.

Table III contains the average breakdown potential and standard deviation for the five different alloys for the as received, high strain fatigue, short-term soak, low strain fatigue, and long-term soak conditions. If breakdown did not occur, breakdown potential was considered as 1000 mV for analysis purposes. With the exception of the BO nitinol wire, all the wires showed high corrosion resistance across all conditions. In fact, the BO nitinol wire exhibited breakdown under every test condition, which allows the change in breakdown potential for the black oxide wire to be tracked across all test conditions. Figure 2 shows representative potentiodynamic curves (for clarity it only shows the forward scan) from each test condition of the BO nitinol wire. The as received BO wire has a very similar curve to both the high strain fatigue and short term soak samples, while the low strain fatigue and long-term soak conditions have noticeably higher potentials and lower currents.

While not as noticeable as the BO nitinol wire, the other four alloys exhibited similar behavior across the test conditions. The values commonly utilized from potentiodynamic curves are the breakdown potential (E_b) and rest potential (E_r), with the over potential calculated as the difference between the breakdown potential and the rest potential ($E_b - E_r$). Figure 3 illustrates the change in the rest potential for all five alloys across all test conditions. While the as received rest potential varies for all alloys, the rest potential appears to converge to zero after 8 days of exposure to PBS, and this was observed in both the low strain fatigue and long-term soak conditions. Figure 4 illustrates the change in breakdown potential (censored at 1000 mV) for all five alloys across all test conditions. With the exception of the one outlier in the short-term soak condition for the MP nitinol, all but the BO nitinol wire had consistently high (>800 mV) breakdown potential. The BO nitinol wire showed a large increase in the breakdown potential for both the low strain and long term soak conditions over the as received wire. Figure 5 plots $E_b - E_r$ for each alloy across the

five test conditions. The MP35N and EP nitinol converged to 1000 mV with increasing time in PBS while the other three materials remained fairly unchanged across test conditions. The statistical results (p -values) for this analysis can be found in Table IV. Figure 6 shows SEM images of specimens in the as received condition and after potentiodynamic testing per ASTM F2129; EP nitinol is not included in Figure 6 because it did not experience breakdown or any surface modification.

With the exception of the BO nitinol wire, no comparison between the fatigue/soak control or between the fatigue/as received condition showed a statistically significant difference in breakdown potential. For the BO nitinol wire, there was a statistical difference in the breakdown for the high strain fatigue/short term soak comparison ($p = 0.009$) with the short-term soak having a greater breakdown potential and for the low strain fatigue/as received comparison ($p < 0.001$) with the low strain fatigue having a greater breakdown potential. In statistical comparisons for MP35N high strain and low strain fatigue to as received and EP nitinol low strain fatigue to long term soak there was a statistical difference in the rest potential that carried over to the $E_b - E_r$ calculation.

DISCUSSION

In total, these results have provided evidence that fatigue testing alone does not reduce the pitting corrosion resistance of the investigated alloys. In Figure 7, SEM images reveal surface damage as a result of the high strain fatigue test condition on wires that were subjected to fatigue testing only and not corrosion testing. These images confirm that we were able to induce surface damage thought to reduce corrosion resistance. However, the high strain test conditions, which are well beyond the expected *in vivo* strains for medical devices,¹⁵ and the short fatigue test duration (less than 8 min) are not representative of physiological conditions. This led to the low strain test condition which was more representative of expected *in vivo* strains. Running this test to 40 million cycles over the course of 8 days was thought to be more physiologically relevant¹⁶ while not taking excessively long. The matching soak conditions, for both high and low strain conditions, were chosen to separate the mechanical effect of fatigue testing from passive surface modification from soaking the wire in PBS.

The alloys we selected were intended to be illustrative of materials with low, medium, and high pitting corrosion resistance per ASTM F2129. Therefore, we expected that 316 LVM and the MP nitinol wires would exhibit more breakdowns before 1000 mV. However, since the 316 LVM and MP nitinol had better pitting corrosion resistance than anticipated, we did not evaluate a material with a medium corrosion resistance per ASTM F2129. Nevertheless, the low breakdown potential of the BO nitinol wire allowed the effect on the breakdown potential to be observed for all test conditions.

Figure 2 shows that there is not a clear difference in the potentiodynamic curves between the high strain fatigue, short term soak, and as received conditions for the BO nitinol wire; this was similarly true for the other materials although only the black oxide nitinol curves are shown as a representative sample. The similarity among these three conditions seems reasonable given that the immersion time in PBS for the high strain fatigue and short term

soak conditions (141 s in the case of black oxide) is short in comparison to the 60-min open circuit potential component of ASTM F2129 and since it appears that fatigue testing does not greatly affect the potentiodynamic response. The low strain fatigue and long term soak specimens possessed a much higher rest potential and had different curves from the other three test conditions, as seen in Figure 2. Since both of those test conditions involved an 8-day soak in PBS at 37°C, it was expected that the surface chemistry would have equilibrated to a PBS environment.¹⁶ Along with having similar rest potential, the shape of the curve is similar for both the low strain fatigue and long term soak specimens further suggesting that the fatigue itself does not significantly alter the cyclic potentiodynamic performance over an 8-day soak in PBS. This is additionally reinforced in Figure 3–5, where there is not a noticeable difference in E_r , E_b , and $E_b - E_r$ between the fatigue and corresponding PBS soak conditions. Increasing the PBS soak time appears to increase the breakdown potential for BO nitinol while bringing the rest potential to 0 mV vs. SCE. In terms of $E_b - E_r$, this value appears consistent for black oxide nitinol, meaning the increase in the breakdown potential is roughly the same as the increase in rest potential. For MP35N, $E_b - E_r$ was observed to decrease with increasing soak time as the value used for breakdown potential stayed the same and the rest potential increased to zero. In the context of medical device testing, ASTM F2129 recommends a minimum vertex potential of 800 mV because potentials are not expected to exceed this value *in vivo*. For our study, we chose a slightly more conservative vertex potential of 1000 mV. Thus, specimens that did not exhibit breakdown during our testing would be expected to not show pitting corrosion under *in vivo* conditions. Since the data is censored, the true $E_b - E_r$ trend could be different from what we observed. However, we believe what is relevant for medical device testing is the conclusion that the breakdown potential for MP35N is above what would be expected *in vivo*. The MP35N material also showed a significant difference ($p < 0.001$) in the rest potential between both the as received test condition and the high strain fatigue test condition and the as received test condition and the low strain fatigue test condition. In both cases the fatigued condition rest potential was greater than the as received rest potential. While MP35N had the longest soak time in PBS for this condition (see Table I), the seven and a half minute soak time was not expected to have an effect on the cyclic potentiodynamic polarization testing as the test has a 60 minute open circuit potential step before the potentiodynamic test segment.

As seen in Table IV, in a preponderance of test conditions there is no statistical difference in the censored breakdown potential between the fatigued condition and the corresponding PBS soak condition. While the 316 LVM high strain fatigue and short term soak comparison did not indicate a significant difference between the results, it is worth noting that two of the high strain fatigued wires exhibited breakdown below 1000 mV while none of the short term PBS soak samples did. Pooling this data with the low strain fatigue and the long term PBS soak conditions for all 316 LVM wires, five of the twelve fatigued wires exhibited breakdown below 1000 mV while none of the twelve PBS soak specimens exhibited breakdown. While this might be indicative of a negative effect of fatigue on the corrosion performance, this is countered by the two breakdowns observed in the MP nitinol wire in the short term soak group when no breakdowns were observed in the high strain fatigue specimens. For the mechanically polished nitinol two of the PBS soak group specimens exhibited breakdown while none of the fatigue group specimens did. All of this together

indicates that in addition to the analysis of breakdown potential showing negligible difference among different test conditions, the number of breakdowns between the fatigue and corresponding PBS soak conditions are also similar and thus provides further evidence that fatigue had no effect on the corrosion behavior.

Considering the number of samples that experienced breakdown was not a concern in analyzing the BO nitinol results since all specimens exhibited breakdown before 1000 mV for every test condition. While in Figure 4 there does not appear to be a noticeable difference in the breakdown potential for the BO nitinol at the high strain fatigue and short-term soak conditions, Table IV does indicate a statistically significant difference in breakdown potential between these two conditions. While this could indicate an effect of fatigue on the corrosion resistance, Table III reveals that both of these test conditions had relatively small standard deviations suggesting that although the p values demonstrates statistical significance, the practical significance of the difference in breakdown potential is limited. Table IV also shows that there is not a significant difference between the as received breakdown potential and the high strain fatigue breakdown potential which implies that the high strain fatigue is not affecting the breakdown potential of the BO nitinol wire. In fact, Table IV shows that for all materials there is not a significant difference between the breakdown potential of the as received specimens and the high strain fatigued specimens. In terms of the breakdown potential, these results indicate that there is no significant effect on the corrosion resistance of these materials from fatigue testing.

CONCLUSIONS

This work set out to establish the effect that fatigue has on the corrosion susceptibility of 316 LVM stainless steel, MP35N cobalt chromium, electropolished nitinol, mechanically polished nitinol, and black oxide nitinol; however this study did not address the effect of fretting or overlapped fatigue conditions and the results should not be extrapolated to those conditions. The results from cyclic potentiodynamic corrosion testing showed that there was generally no significant difference in corrosion susceptibility (as measured by breakdown potential) between the fatigued wire and the wire soaked in PBS for the same period of time as the fatigue test. Since SEM images confirmed that there was surface damage after the high strain fatigue was applied, we can be fairly certain that these cracks did not increase the corrosion susceptibility of the alloys tested.

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FIGURE 1.
Rotary bend fatigue test set up.

FIGURE 2.

Representative curves showing how test condition affects potentiodynamic response. The black oxide wire is shown since it consistently broke down across all conditions.

FIGURE 3.

Composite plot of the rest potential (E_r) for all alloys and test conditions ($n = 6$ for each condition).

FIGURE 4.

Composite plot of the breakdown potential (E_b) for all alloys and test conditions ($n = 6$ for each condition). With the exception of BO nitinol, each material and condition contains censored data when no breakdown occurred and E_b was assumed to be 1000 mV.

FIGURE 5.

Composite plot of the over potential ($E_b - E_r$) for all alloys and test conditions ($n = 6$ for each condition). With the exception of BO nitinol, each material and condition contains censored data when no breakdown occurred and E_b was assumed to be 1000 mV.

FIGURE 6.

Scanning electron microscopy of as received wire surfaces and post-corrosion testing. A – D are the as received surfaces for 316 LVM, MP35N, MP nitinol, and BO nitinol while E – H show evidence of corrosion or surface modification for the 316 LVM, MP35N, MP nitinol, and BO Nitinol wires. EP nitinol was excluded from this figure as it did not experience breakdown or surface modification during the cyclic potentiodynamic tests.

FIGURE 7.

Scanning electron microscopy of the as received wire surface and post-high strain fatigue testing. A – C are the as received surfaces for 316 LVM, MP35N, and MP nitinol while D – F show evidence of crack initiations along the surface for 316 LVM, MP35N, and MP nitinol respectively.

TABLE I

Rotary Bend Fatigue Test Parameters

Alloy	Wire Diameter (mm)	Low Strain			High Strain		
		ϵ (%)	Cycles (#)	Test Duration	ϵ (%)	Cycles (#)	Test Duration
316 LVM	0.178	0.14	40,000,000	8 days	0.50	13,475	3 m 45 s
MP35N	0.178	0.14	40,000,000	8 days	0.50	25,564	7 m 6 s
EP NiTi	0.500	0.40	40,000,000	8 days	1.00	13,287	3 m 41 s
MP NiTi	0.500	0.40	40,000,000	8 days	1.00	9,105	2 m 32 s
BO NiTi	0.500	0.40	40,000,000	8 days	1.00	8,113	2 m 15 s

TABLE II

Results of Preliminary High Strain Fatigue Testing (n = 4 for Each Condition)

Alloy	ϵ (%)	Mean Cycles to Failure	Standard Deviation
316 LVM	0.50	14,184	801
MP35N	0.50	26,910	1,472
EP nitinol	1.00	13,986	779
MP nitinol	1.00	9,585	609
BO nitinol	1.00	8,541	527

TABLE III

Average Breakdown Potential and Standard Deviation (mV) per Alloy and Condition (n = 6 for Each Condition)

	As Received	High Strain Fatigue	Short-Term Soak	Low Strain Fatigue	Long-Term Soak
316 LVM	972 ± 68	950 ± 93	1000 ± 0	945 ± 61	1000 ± 0
MP35N	1000 ± 0	1000 ± 0	1000 ± 0	1000 ± 0	939 ± 149
EP nitinol	1000 ± 0	1000 ± 0	1000 ± 0	1000 ± 0	1000 ± 0
MP nitinol	928 ± 175	1000 ± 0	784 ± 354	1000 ± 0	1000 ± 0
BO nitinol	-52 ± 9	-57 ± 5	-39 ± 10	460 ± 27	428 ± 61

With the exception of BO nitinol, each material, and condition contains censored data when no breakdown occurred and breakdown potential was assumed to be 1000 mV.

TABLE IV

p-Values from Statistical Analyses of the Cyclic Potentiodynamic Test Results in Terms of the Effect of Fatigue Between the Relevant Soak Condition or the as Received Wire

		Low Strain Fatigue and Long Term Soak	High Strain Fatigue and Short Term Soak	Low Strain Fatigue and As Received	High Strain Fatigue and As Received
316 LVM	E_t	0.295	0.712	0.490	0.660
	E_b	0.055	0.138	0.311	0.528
	$E_b - E_t$	0.571	0.178	0.561	0.174
MP35N	E_t	0.290	0.774	< 0.001*	< 0.001*
	E_b	0.317	N/A	N/A	N/A
	$E_b - E_t$	0.112	0.429	0.001*	0.001*
EP nitinol	E_t	0.011*	0.049	0.353	0.302
	E_b	N/A	N/A	N/A	N/A
	$E_b - E_t$	0.005*	0.016	0.145	0.982
MP nitinol	E_t	0.782	0.442	0.269	0.067
	E_b	N/A	0.138	0.317	0.317
	$E_b - E_t$	0.765	0.563	0.870	0.019
BO nitinol	E_t	0.246	0.190	0.003*	0.001*
	E_b	0.279	0.009*	< 0.001*	0.357
	$E_b - E_t$	0.191	0.010*	0.390	0.004*

Rest potential, E_r , for all materials was compared with two-sample t -tests and $\alpha = 0.0125$. Breakdown potential, E_b , and over potential, $E_b - E_r$, for BO nitinol were also compared with two-sample t -tests and $\alpha = 0.0125$. E_b and $E_b - E_r$ for all other materials were analyzed with log-rank tests and $\alpha = 0.0125$. Statistically significant differences are marked by *.