Corrosion Resistance of Composite NiTi/Platinum wires

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Prepared by:

Thomas Gonchar, BSEng

Reviewed by:

Christine Trepanier, MScA

Alan Pelton, PhD

NDC
Nitinol Devices & Components

47533 Westinghouse Dr
Fremont, CA 94536
510/623-6996 phone
510/623-6808 fax

1.0 BACKGROUND

This report presents results from corrosion testing of composite NiTi/Pt wires in Hank's physiological solution.

Corrosion of implant devices can have deleterious effects on the device performance or may result in the release of corrosion products with harmful biological consequences. Therefore, it is important to determine the general corrosion behavior of such devices as well as their susceptibility to localized corrosion.

The corrosion resistance of implant devices is assessed by performing potentiodynamic polarization tests on the implant while it is immersed in an appropriate artificial physiological solution (Hank's solution in this study). Based on the shape of the resulting polarization curve it is possible to determine both the general corrosion behavior of an implant device as well as its ability to resist localized corrosion (pitting). It is important to note that this test method is designed to reach conditions that are severe enough to cause pitting of the material and that these conditions may not necessarily be encountered *in vivo*. Still, if the device is highly resistant to corrosion, pitting may not occur despite the severity of the test conditions. A breakdown potential, corresponding to the potential at which the oxygen evolution reaction begins to occur, may still be defined in such cases.

2.0 REFERENCES

2.1 ASTM F2129 "Standard Test method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices"
2.2 NDC Technical Laboratory Notebook #1257
2.3 NDC Technical Laboratory Notebook #1264

3.0 EQUIPMENT and MATERIALS

3.1 Potentiostat, EG&G Princeton Applied Research model 273A N-152 (calibration due 11/26/03)
3.2 Potentiostat, EG&G Princeton Applied Research model 273A N-171 (calibration due 11/27/03)
3.3 pH meter, Acumet portable, Fisher Scientific, model AP61, AA1293 (calibration due 02/25/04)
3.4 Temperature controlled water bath, VWR and thermocouple Barnant, AE7659 (calibration due 06/09/04)
3.5 Hank's solution: Hank's balanced salts, Sigma H-6136, Lot 102K83101, exp. 11/05; Sodium Bicarbonate powder, JT Baker 3506-01, Lot V11H01; distilled water.
3.6 Nitrogen gas, high purity
3.7 Gas flow gauges, Gilmont Instruments model #65mm, PMC0125 (calibration due 04/23/04)

4.0 METHOD

Three groups of wires; two composed of NiTi/Pt composite wire samples, and one composed of pure NiTi wire samples, were tested according to ASTM F2129, "Standard Test Method for Conducting Cyclic Potentiodynamic Polarization Measurements to Determine the Corrosion Susceptibility of Small Implant Devices."

The composite wires consisted of a nitinol tube with a platinum core. The platinum core represented either 10% or 30% of the wire. The third group of wires was nitinol and was used as a control. Prior to corrosion testing, all wires were electropolished to enhance their corrosion resistance.

In accordance with ASTM F2129, an EG&G Princeton Applied Research potentiostat model 273A is used to conduct the potentiodynamic polarization corrosion test. The potentiostat is controlled by a computer with Electrochemistry PowerSuite corrosion test software. A saturated calomel electrode (SCE) is used as a reference electrode for the potential. Two platinum auxiliary electrodes are used as counter electrodes. Testing is conducted in an appropriate polarization cell as recommended in ASTM F2129 at a potential scan rate of 0.167 mV/sec. A water bath is used to maintain the test solution (Hank's solution) temperature at 37 ± 1°C.

Hank's solution (pH of 7.4±0.1) was mixed according to the manufacturer's instructions: Hank's balanced salts were mixed with 0.35g/L of sodium bicarbonate and distilled water. Nitrogen gas purge was conducted starting 30 minutes prior to testing and throughout the test.

To minimize the damage to the test specimen and allow identification of the pit initiation site, the reverse polarization scan was not performed during these tests. The tests were stopped once the breakdown potential was reached. Identification of the pit initiation site allows determination of the cause of pit initiation and can be used to optimize manufacturing processes to improve corrosion resistance. If the implant undergoes extensive damage during the reverse polarization scan, it may not be possible to identify the pit initiation site and therefore the information cannot be used to improve manufacturing processes.

The corrosion resistance of the wires was characterized in terms of their corrosion potential (E_{corr}), corrosion current density (I_{corr}), breakdown potential (E_{bd}), and corrosion rate (CR).

5.0 RESULTS AND DISCUSSION

The results for the different corrosion parameters are summarized in Table I on the next page. The corrosion potential and polarization curves are presented in Appendix I in Figures 1.1 through 1.6.

The I_{corr} value gives an indication of the corrosion rate of the device. The average value of I_{corr} for each group is presented in Table 1 on the next page. The corrosion rates presented in Table 1 are based on a density of 6.45 g/cm^3, an equivalent weight of 17.8 g, and a test surface area of 0.5 cm^2. The equivalent weight and density values for pure nitinol were used for all of the wires since only a small amount (less than 10%) of the exposed test surface of the composite wires was platinum. Values of I_{corr} did not vary significantly from one group to the next.

Table I. Corrosion results for NiTi and NiTi/Pt wires tested in Hank's solution

Group	Sample	E_{corr} mV vs SCE	I_{corr} nA/cm^2	E_{bd} mV vs SCE	CR mmPY	Final pH*
NiTi	1	-447	19.5	910	1.8E-4	8.7
	2	-432	6.8	933	6.1E-5	8.7
	3	-474	4.6	982	4.1E-5	8.4
Average ± St. Dev.		**- 451 ± 21**	**10.3 ± 8.1**	**942 ± 37**	**9.3E-5 ± 7.3E-5**	**8.6 ± 0.1**
NiTi +10% Pt	3	-436	6.7	958	6.1E-5	8.4
	4	-468	4.3	867	3.9E-5	8.6
	5	-406	4.9	890	4.4E-5	8.5
Average ± St. Dev.		**- 437 ± 31**	**5.3 ± 1.3**	**905 ± 47**	**4.8E-5 ± 1.1E-5**	**8.5 ± 0.1**
NiTi +30% Pt	1	-460	4.2	875	3.8E-5	8.7
	2	-456	12.1	927	1.1E-4	8.4
	3	-429	7.3	995	6.6E-5	8.4
Average ± St. Dev.		**- 448 ± 17**	**7.9 ± 4.0**	**932 ± 60**	**7.1E-5 ± 3.6E-5**	**8.5 ± 0.2**

*Deaeration causes a pH increase because of displacement of CO_2 from solution.

The breakdown potential (E_{bd}) is a measure of the resistance of the device to localized corrosion. E_{bd} values did not vary significantly between groups. Values of corrosion potential (E_{corr}) also did not vary significantly between wire groups.

Visual inspection of the devices after testing did not reveal any localized corrosion (pitting) on any of the samples. Furthermore, no evidence of galvanic corrosion was observed on any of the composite wires which may be attributed to the small surface area ratio of exposed Pt/NiTi and to the highly corrosion resistant layer on NiTi created by the electropolishing process. Post-corrosion testing images of the end of NiTi + 10% sample #4 appear in Appendix II.

6.0 CONCLUSIONS

This memo reports the results from corrosion testing of NiTi and NiTi/Pt composite wires in Hank's physiological solution.

Based on the results from this study:

1. Corrosion behavior did not vary significantly between the wire groups.

2. No evidence of galvanic corrosion was observed on any of the electropolsihed composite NiTi/Pt wires which may be attributed to the small surface area ratio of exposed Pt/NiTi and to the optimization of the NiTi's corrosion resistance through electropolishing.

APPENDIX I

CORROSION POTENTIAL AND POLARIZATION CURVES FOR NITINOL AND NITINOL/PLATINUM WIRES

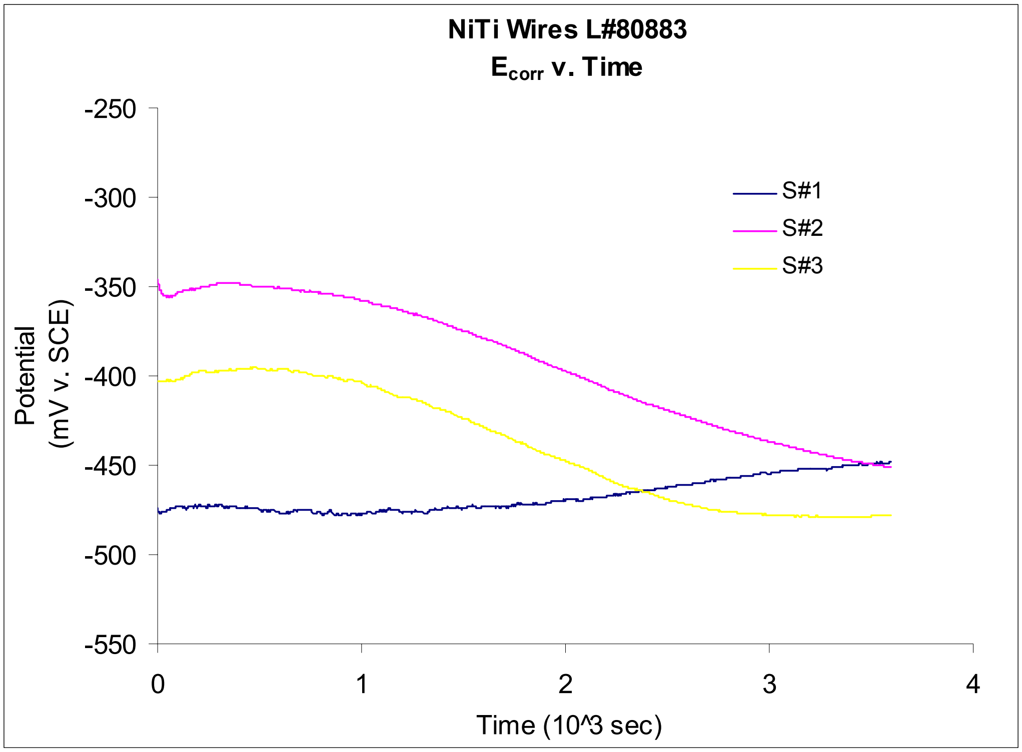

Figure 1.1 E_{corr} vs time for nitinol wires (L#80883)

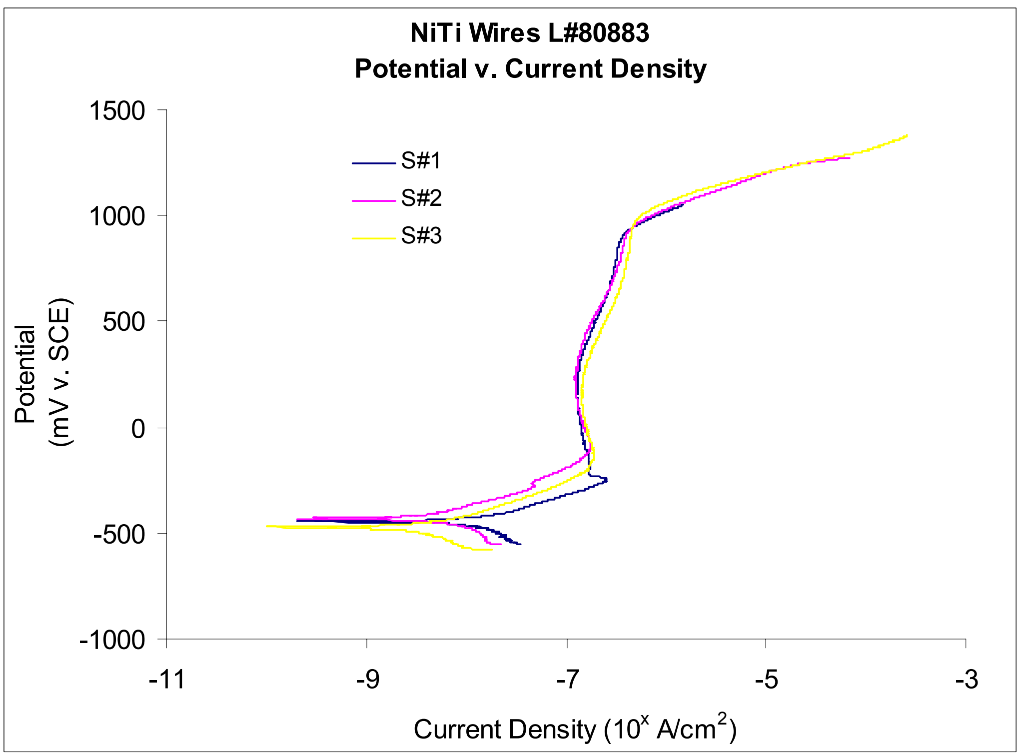

Figure 1.2 Polarization curves for nitinol wires (L#80883)

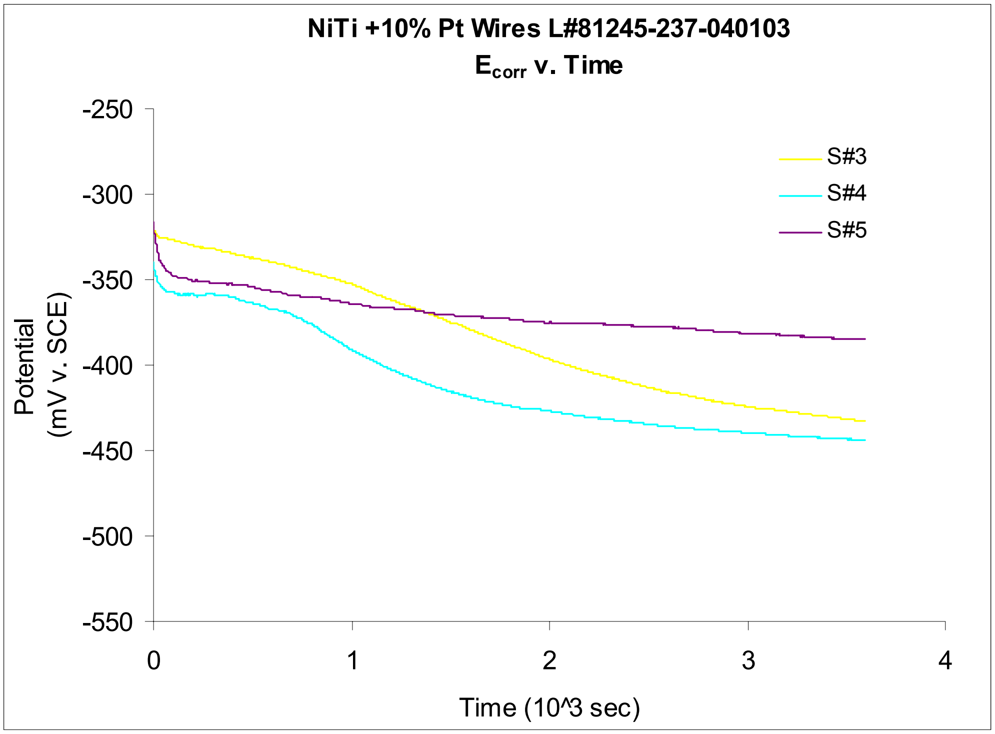

Figure 1.3 E_{corr} vs time for nitinol + 10% platinum wires (L#81245-237-040103)

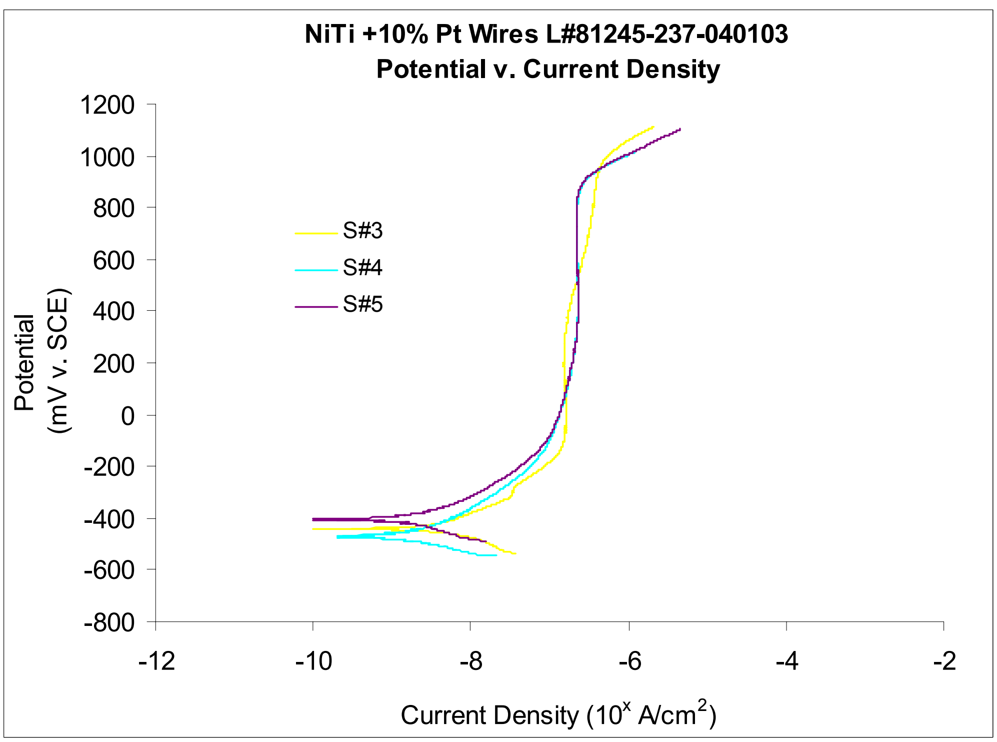

Figure 1.4 Polarization curves for nitinol + 10% platinum wires (L#81245-237-040103)

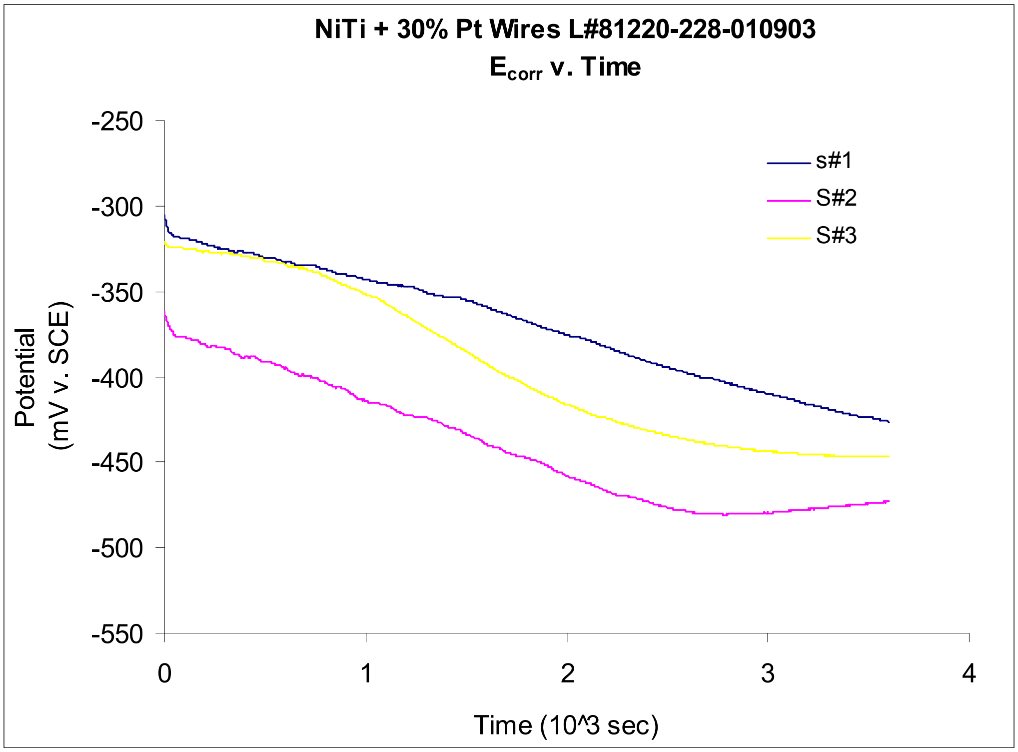

Figure 1.5 E_{corr} vs time for nitinol + 30% platinum wires (L#81220-228-010903)

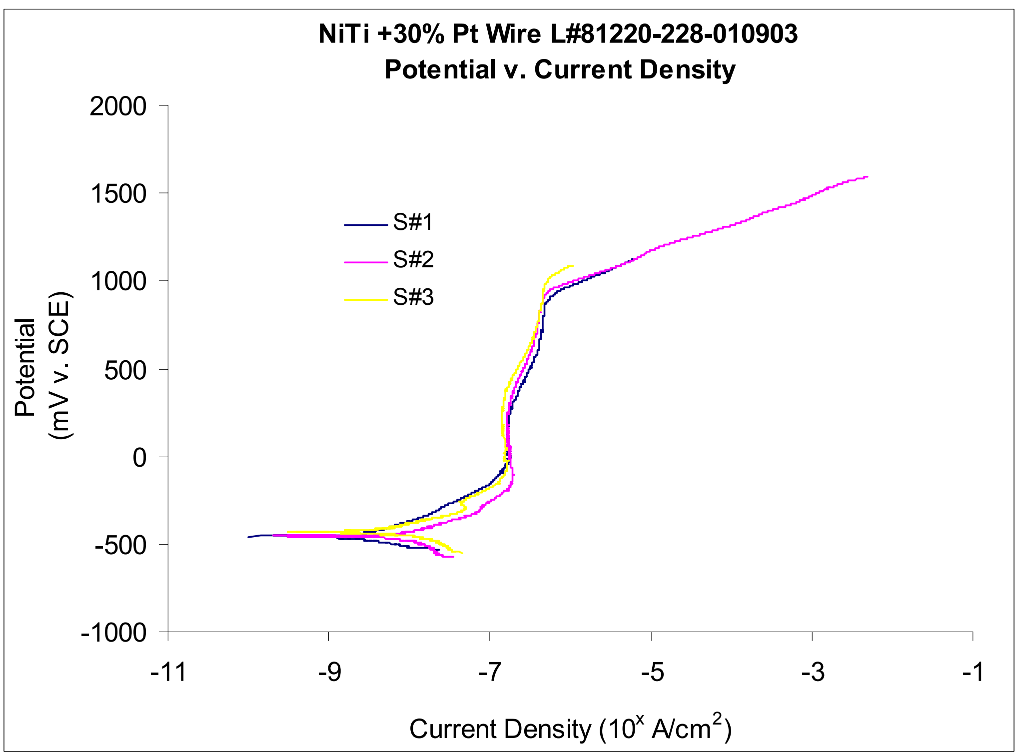

Figure 1.6 Polarization curves for nitinol + 30% platinum wires (L#81220-228-010109)

APPENDIX II

POST-CORROSION IMAGES
OF NITINOL/PLATINUM WIRE

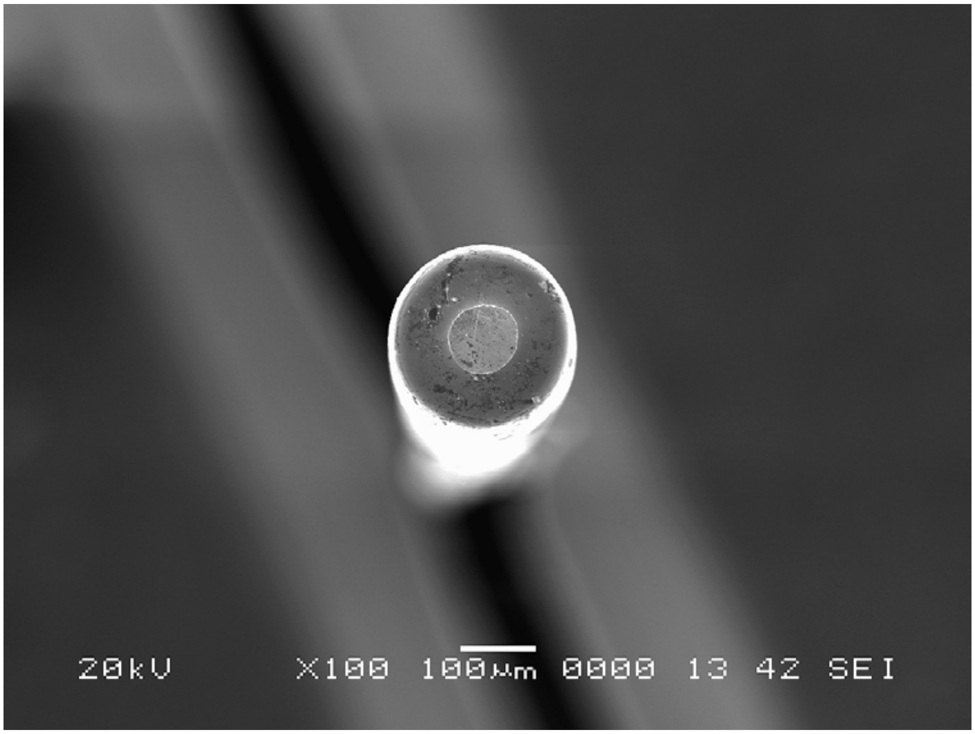

Figure 2.1 Post-corrosion image of the exposed end of NiTi + 10% Pt wire sample #4

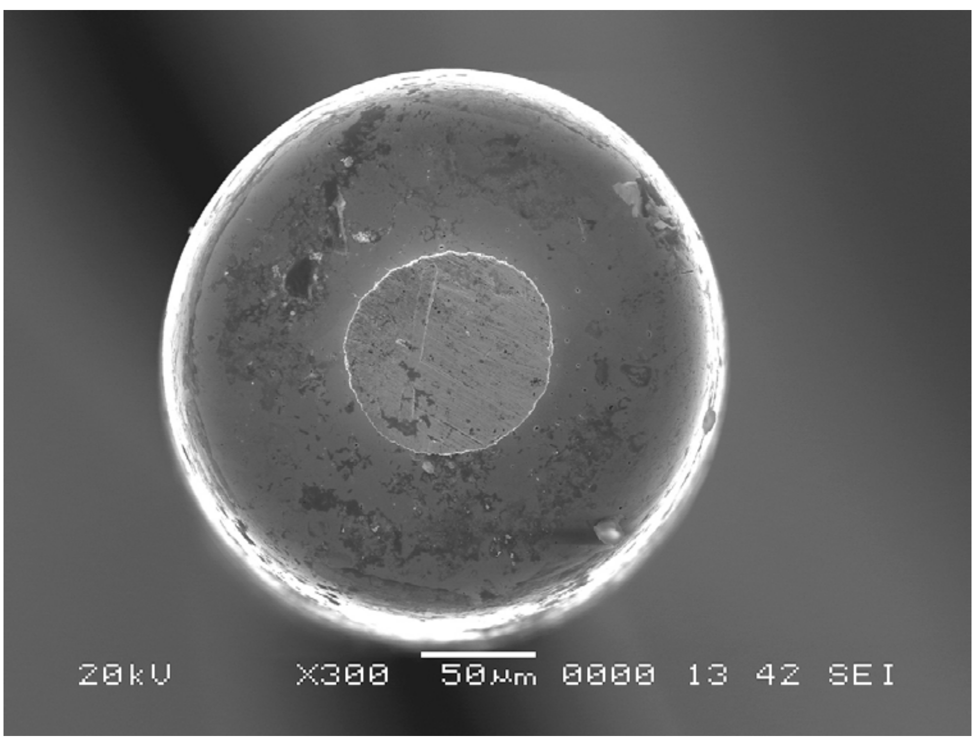

Figure 2.2 Higher magnification image of the exposed end of NiTi + 10% Pt wire sample #4 post-corrosion testing