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Topographical and Compositional Homogeneity of Electropolished NiTi Alloy Surfaces

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Introduction

Nickel-Titanium (NiTi) alloy exhibits a number of unique properties, including shape memory and superelasticity, that make it suitable for use in endovascular devices [1]. However, the high nickel-content (~54 weight %) remains a concern with reference to nickel cytotoxicity. The corrosion resistance of NiTi can be significantly improved (thereby reducing nickel leaching) by surface treatment methods such as electropolishing, air aging, heat treatment, and nitric acid passivation. Of these treatments, electropolishing generates superior corrosion resistance [2]. This superior corrosion resistance has been attributed to the thickness of the surface oxide, the resulting surface topography and chemistry. The objective of this study is to understand the effect of EP on surface topography and chemistry of NiTi alloy surfaces.

Materials and Methods

NiTi disc samples were wet-polished using SiC paper to 1200 grit finish (MP-NiTi). These samples were electropolished by NDC:CORDIS (Fremont, CA) to meet ASTM F86 specifications (EP-NiTi). Potentiodynamic polarization testing was conducted per ASTM F2129-01 in de-aerated Hank's balanced salt solution (HBSS) at 37°C. X-ray photoelectron spectroscopy (XPS) characterization was performed using a Kratos AXIS 165 Multi-technique Electron Spectrometer. Survey scans and high resolution scans of C1s, O1s, Ti2p, and Ni2p(3/2) were obtained, peak-fit and analyzed for surface composition/chemistry. Scanning electron microscopy (SEM) was conducted to obtain scout images. Atomic force microscopy (AFM) was then performed using a PicoSPM (Molecular Imaging, Phoenix, AZ) scanning probe microscope. Topographical maps were obtained at 6x6 µm and 3x3 µm scan sizes.

Results and Discussion

The corrosion data obtained by polarization testing indicated that the electropolished samples exhibited a low corrosion current density (units nA/cm²) and high breakdown potential (~950 mV vs. SCE) compared to the mechanically polished sample. This result confirmed the presence of a protective passive film on the surface of electropolished samples [3]. Composition data obtained from XPS analysis is summarized in Table 1.

Table 1: Relative atomic concentrations and concentration ratio as determined by XPS. Values are average of n=3.

Sample Type	% Atomic Mass				O/Ti Ratio
	Ni 2p	O 1s	Ti 2p	C 1s	
EP-NiTi	1.5	59.4	15.2	23.9	3.69
MP-NiTi	8.7	50.6	13.8	26.8	3.90

In general, the amount of Ni on the surface was significantly reduced and a homogeneous titanium oxide was observed on the EP-NiTi samples. Preliminary stoichiometric analysis indicates the presence of mostly TiO₂ on the EP-NiTi samples, while the MP-NiTi samples had inclusions of sub-oxides.

Initial SEM images indicated that the EP-NiTi samples had a wavy and rougher surface compared to the mechanically polished samples. However, AFM surface maps at a µm-scale indicated that EP-NiTi samples exhibited a smoother and uniform surface. Consolidated roughness parameters obtained from analysis of the AFM surface maps at both scan sizes are presented in Table 2.

Table 2: Roughness parameters as determined by AFM. Values are average of n=3.

Sample Type	6x6 µm Scan		3x3 µm Scan	
	R _a (Å)	R _{p-v} (Å)	R _a (Å)	R _{p-v} (Å)
EP-NiTi	15	320	8	206
MP-NiTi	98	1092	44	329

While the electropolishing process may have contributed to an overall wavy surface appearance, the peak-to-valley distances are significantly lower for the EP-NiTi samples. As electropolishing is a process that relies on preferential removal of material to obtain the desired surface finish, this observation is justified. Preliminary Scanning Vibrating Probe analysis of sample surfaces is also indicating that this topographical and chemical homogeneity on EP-NiTi samples results in a more uniform distribution of electrochemical behavior.

Conclusions

Electropolished NiTi samples exhibited a more homogenous topographical and compositional surface that contributed to their enhanced corrosion behavior. While the electropolished NiTi samples exhibited a wavy appearance at a macro-scale, micron-scale analysis indicated that the surfaces are smoother than mechanically polished surfaces.

References

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