

STRUCTURE AND PROPERTIES OF OXYGEN IMPLANTED NiTi SURFACES

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Motivation

Shape memory and superelastic NiTi alloy:

Excellent biocompatibility is due to **native TiO₂ surface layer** In some cases **corrosion in vivo** is demonstrated

- Medical applications:
- Vascular stents
 - Vena cava filters
 - Orthopedic implants ...
- High concentration of Ni (~50 at%):
- ⊗ Allergic
 - ⊗ Toxic
 - ⊗ Carcinogenic

Objective

A corrosion resistant NiTi surface without nickel release is demanded.

Plasma immersion ion implantation (PIII) (by O⁺ ions)



NiTi surface

- Formation of Ni depleted surface layer
- Increase of the surface stability
- Barrier creation against the Ni release from NiTi
- No change of bulk properties

◆ Implanted NiTi: Microstructure, Morphology, Properties

Experimental

Ion implantation

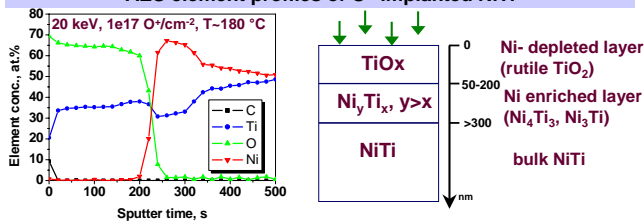
- Substrates:** NiTi alloy (55.90 wt.%Ni, 44.08 wt.% Ti) from Memory-Metalle,
- Plasma Ion Implantation:**
- RF-discharge as a plasma source
 - high voltage pulses: H = 100-600 Hz, τ = 5 μs
 - ion energy of 10 - 20 keV
 - ion fluences of (1-10) × 10¹⁷ cm⁻²
 - implantation temperature 50-250 °C

Analysis

- Atomic force microscopy (AFM)
- Auger electron spectroscopy (AES)
- Elastic recoil detection analysis (ERDA)
- Corrosion measurements
- Scanning electron microscopy (SEM)
- Spectroscopic ellipsometry (SE)
- X-ray diffraction analysis
- Electrochemical analysis
- Biocompatibility tests

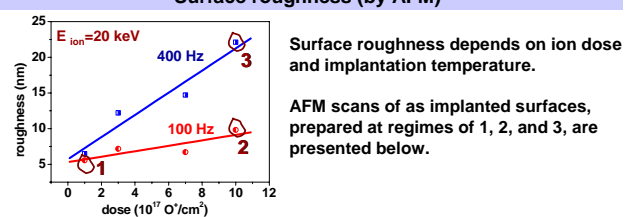
Element depth profiles

AES element profiles of O⁺-implanted NiTi



Roughness & morphology

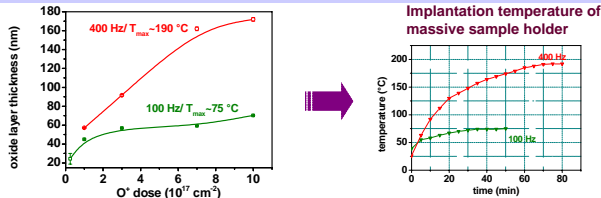
Surface roughness (by AFM)



Surface roughness depends on ion dose and implantation temperature.

AFM scans of as implanted surfaces, prepared at regimes of 1, 2, and 3, are presented below.

Oxide layer thickness (by spectroscopic ellipsometry)



- Ni depleted oxide layer is transparent rutile TiO₂
- TiO₂ layer thickness depends on ion dose and temperature (E_{ion}=const)
- Oxygen transport ⇒ diffusion mechanism

AFM scans of O⁺-implanted surfaces (1x1 μm)

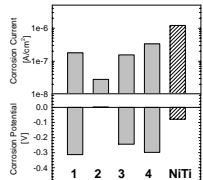


At higher doses and temperatures a formation of surface aggregates is shown. Probably these are crystallites of titanium oxide.

Properties

Corrosion test

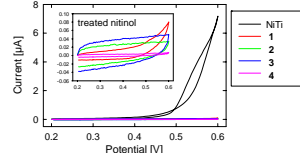
By potentiodynamic polarization (10 mV/s) in Ringer's solution



All of O⁺ Implanted NiTi have a much lower corrosion current density.

Catalytic activity of Ni

for glucose oxidation (5 mmol/L) (The cyclic voltammograms)



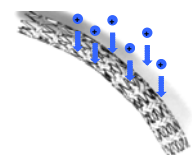
All of O⁺ Implanted NiTi show no catalytic activity for the oxidation of glucose, which would be characteristic for nickel.

Biocompatibility: hemocompatibility and cell culture test

- O⁺ Implanted NiTi surfaces:
 - adsorb less fibrinogen
 - activate the contact system less than the NiTi
 - have higher conformation changes of fibrinogen
- No difference between O⁺ implanted NiTi and untreated NiTi was found for the platelet adherence, endothelial cell activity.

Summary

- Oxygen PII implantation is a useful method to decrease the nickel concentration in the subsurface layer of NiTi alloy for cardiovascular application.
- O⁺ implanted NiTi has three layers: TiO₂ surface layer, Ni enriched area, bulk NiTi.
- The titanium oxide surface prevents corrosion and out-diffusion of Ni ions.
- Thickness, morphology and properties are conditioned in general by implantation temperature.



Acknowledgments

Dr. H. Reuther (FZR) for AES measurements, Dr. M. Vinnichenko for SE studies.

This study was financially supported by Boston Scientific SCIMED