

# 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 TiO<sub>2</sub> 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<sup>+</sup> 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

## Ion implantation

**Substrates:** **NiTi alloy** (55.90 wt.%Ni, 44.08 wt.% Ti) from Memory-Metalle,

**Plasma Ion**

- RF-discharge as a plasma source

**Implantation:**

- high voltage pulses: H = 100-600 Hz, τ = 5 μs
- ion energy of 10 - 20 keV
- ion fluences of (1-10) × 10<sup>17</sup> cm<sup>-2</sup>
- implantation temperature 50-250 °C

## Experimental

## Analysis

Atomic force microscopy (AFM)

Scanning electron microscopy (SEM)

Auger electron spectroscopy (AES)

Spectroscopic ellipsometry (SE)

Elastic recoil detection analysis (ERDA)

X-ray diffraction analysis

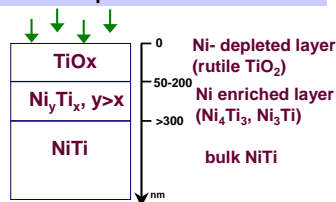
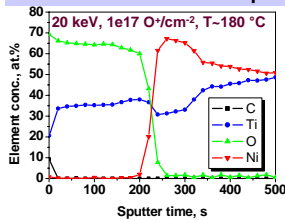
Corrosion measurements

Electrochemical analysis

Biocompatibility tests

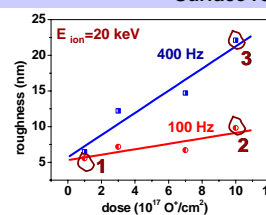
## Element depth profiles

AES element profiles of O<sup>+</sup>-implanted NiTi



## Roughness & morphology

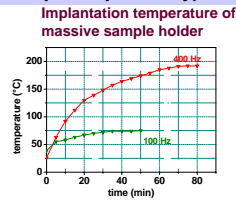
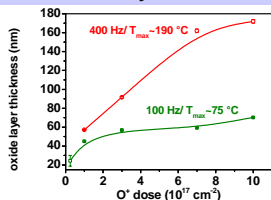
Surface roughness (by AFM)



Surface roughness depends on ion dose and implantation temperature.

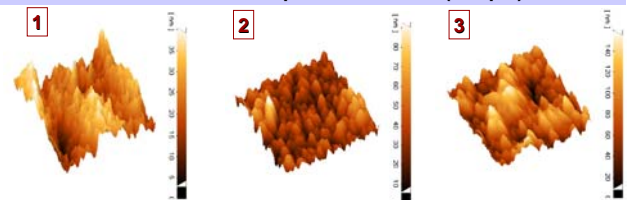
AFM scans of as implanted surfaces, prepared at regimes of 1, 2, and 3, are presented below.

## Oxide layer thickness (by spectroscopic ellipsometry)



- Ni depleted oxide layer is transparent rutile TiO<sub>2</sub>
- TiO<sub>2</sub> layer thickness depends on ion dose and temperature (E<sub>ion</sub>=const)
- Oxygen transport ⇒ diffusion mechanism

## AFM scans of O<sup>+</sup>-implanted surfaces (1x1 μm)

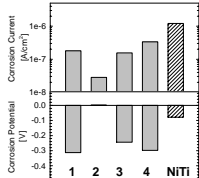


At higher doses and temperatures a formation of surface aggregates is shown. Probably these are crystallites of titanium oxide.

## Properties

### Corrosion test

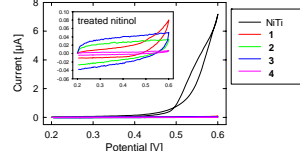
By potentiodynamic polarization (10 mV/s) in Ringer's solution



All of O<sup>+</sup> Implanted NiTi have a much lower corrosion current density.

### Catalytic activity of Ni

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



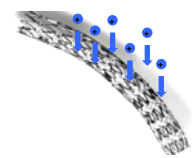
All of O<sup>+</sup> Implanted NiTi show no catalytic activity for the oxidation of glucose, which would be characteristic for nickel.

### Biocompatibility: hemocompatibility and cell culture test

- O<sup>+</sup> Implanted NiTi surfaces:
    - adsorb less fibrinogen
    - activate the contact system less than the NiTi
    - have higher conformation changes of fibrinogen
- No difference between O<sup>+</sup> 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<sup>+</sup> implanted NiTi has three layers: TiO<sub>2</sub> 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.



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