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POROUS SURFACE OF NiTi ALLOY PRODUCED BY PLASMA ION IMPLANTATION

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Motivation

Nanoporous surface layers of TiOx on Ti or NiTi:

- ❑ *Electrochemical or*
- ❑ *Catalytical applications*
- ☑ *Carrier in drug release system*

Interconnected pores with size of few nanometers (~ size of drug molecule)

Objective

Formation of nanoporous surface layers on NiTi (superelastic & memory shape alloy) by high dose He^+ ion implantation:

Plasma immersion ion implantation (PIII) → He^+ → NiTi alloy surface → Microstructure, Morphology, Properties

Experimental

Ion implantation

Substrates: NiTi alloy (55.90 wt.%Ni, 44.08 wt.% Ti) from Memory-Metalle, Ti (purity 99.6) from Goodfellow GmbH – as reference.

Ion energy: 20 keV Temperature: 100 – 400 °C

Ion fluences: (1-10)x10^{17} cm^{-2} Base pressure: <8x10^{-4} Pa

Analysis

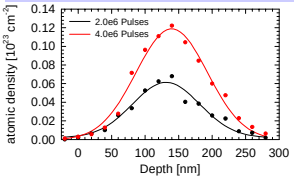
Atomic force microscopy (AFM) Scanning electron microscopy (SEM)

Auger electron spectroscopy (AES) Elastic recoil detection analysis (ERDA)

Transmission electron microscopy (TEM)

Element depth profiles

Helium depth profiles in Ti (ERDA)

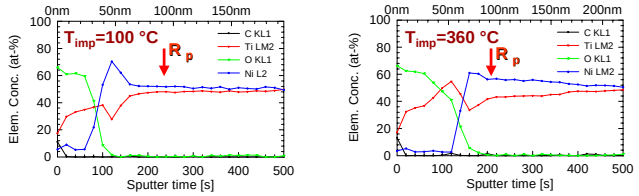


The integral dose of the **2e6 pulses** implantation is **0.8 x 10^{17} cm^{-2}**, for the **4e6 pulses** it is **1.57 x 10^{17} cm^{-2}**. Measured dose < expected implanted dose (~ 5 x 10^{17} cm^{-2}).

Projected Ranges at 20 keV (TRIM):

Ti: 127 nm, NiTi: 92 nm

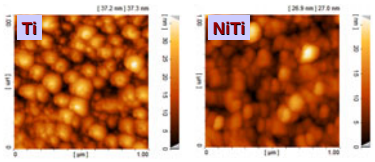
AES element profiles of He^+-implanted NiTi



- Ni depletion oxide layer (oxygen from residual background gas in chamber)
- Ni enriched zone (transport of Ni atoms from oxide surface layer)
- Bulk NiTi

Microstructure & morphology

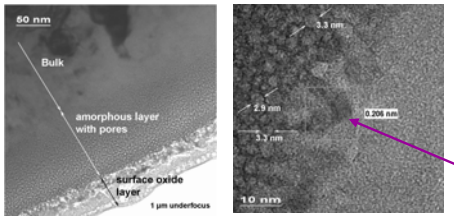
AFM scans of He^+-implanted surfaces (1x1 µm)



Implantation:
20 keV, 4e6 pulses, T~ 100 °C

Morphology of surfaces is very similar for both NiTi and Ti.

TEM images of He^+-implanted NiTi



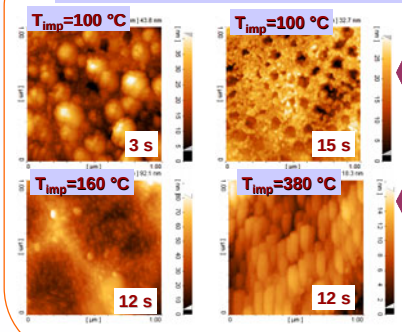
Right: Detailed image of the interface layer between the surface oxide and porous layer.

Fine crystallite with d=0.206 nm (perhaps Ni_4Ti_3 (d=0.209 nm))

Left: overview, showing the existence of three layers: surface oxide; porous amorphous layer; bulk.

Morphology after etching in 1%HF

AFM scans (1x1 µm) of implanted surfaces after etching



Implanted Ti (at 100 °C): surface oxide layer was removed and there was access to a deeper porous layer.

Implanted Ti (at >160 °C): porous structure was mainly not visible.

Implanted NiTi (at 100 °C): ? porous structure was not found by AFM after etching for 5s to 40s.

Summary

- Plasma immersion ion implantation of He^+ ions into NiTi lead to formation nanoporous surface layer.
- Helium implanted NiTi shows three layers:
  1. Titanium oxide surface layer (~50 nm);
  2. Amorphous nanoporous NiTi area (~150 nm);
  3. Bulk NiTi alloy.
- By chemical etching of the implanted Ti surfaces a nanoporous layer can be obtained, however for implanted NiTi sufaces such effect was not revealed.

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