APPLICATION OF AN ECR ION SOURCE FOR IONIC FUNCTIONALIZATION OF IMPLANT MATERIALS ON THE NANOSCALE

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## Motivation

<table>
<thead>
<tr>
<th>Target material</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beam</td>
<td>Au</td>
<td>Ca</td>
<td>Si</td>
</tr>
<tr>
<td>Ti</td>
<td>Ti</td>
<td>Ti</td>
<td>ZrO$_2$</td>
</tr>
</tbody>
</table>
Motivation

A.
- GNPs (Gold Nano-Particles) can chemically bond many types of biomolecules.

- Plasmon effect of the GNPs: energy transfer between the implants covered by GNPs and the light with appropriate wavelength may be able to destroy the bacteria molecules around the implants.

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Motivation

B.
The implanted Ca ions may increase and accelerate the adherence of the human tissue due to diffusion.

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Motivation

C.  
- $\text{ZrO}_2$ (non-silica-) based restorations have become very popular in the dentistry (esthetic).
- Silicon implantation in order to bond polymer molecules to the ceramic.

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## Motivation

### Using an ECR Ion Source for ionic implantation:

<table>
<thead>
<tr>
<th>Advantages</th>
<th>Disadvantages</th>
</tr>
</thead>
<tbody>
<tr>
<td>- High gas efficiency</td>
<td>- Relatively high operational costs</td>
</tr>
<tr>
<td>- Plasmas and beams produced from solid</td>
<td>- Inhomogeneity of the beam</td>
</tr>
<tr>
<td>- Beam energy range, implantation depth is ideal</td>
<td>- Using non analyzed beam (not pure)</td>
</tr>
<tr>
<td>- Effect of the charge state of the incident ions</td>
<td></td>
</tr>
</tbody>
</table>
The Atomki ECR Ion Source

- Standard, stand-alone device. No post acceleration.

- Strong advantage is the modularity of the source

<table>
<thead>
<tr>
<th>Specification</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microwave frequency</td>
<td>14.3 GHz (6-18GHz)</td>
</tr>
<tr>
<td>Microwave power</td>
<td>0.1 - 1000 W</td>
</tr>
<tr>
<td>Resonant field</td>
<td>(14.3 GHz) 0.52 Tesla</td>
</tr>
<tr>
<td>Axial magnetic field peak</td>
<td>1.2 Tesla</td>
</tr>
<tr>
<td>Maximum of the radial field (hexapolar)</td>
<td>1.2 Tesla</td>
</tr>
<tr>
<td>Plasma chamber L and ID</td>
<td>5.8 cm / 20 cm</td>
</tr>
<tr>
<td>Extraction voltage</td>
<td>50V - 30 kV</td>
</tr>
</tbody>
</table>
Requirements and difficulties
Requirements and difficulties

- Metallic (gold, calcium and silicon) ion beam is necessary for the planned treatments.
- The implantation depth must be characterized and precisely controlled.
  - Depth profile analysis of the samples made e.g. by SNMS is necessary
  - Theoretical calculation for realistic prediction.
- Clinical tests require a series of the samples (at least 60 paces)
- Relatively big size of the samples (10 mm x 10 mm)
- Inhomogenity of the beam.
- Dose requirements: it must be controlled from $10^{16}$ ion/cm$^2$ up to $10^{18}$ ion/cm$^2$.
- Insulation target e.g. ZrO$_2$
Ion beam developments

Production of gold ion beam:

- Sputtering technic
- Sputtering electrode temperature
- Sputtering electrode: 2 kV
- Support gas: oxygen

Optimized for middle charge states (Us=10 kV)

Optimized for low charge states (Us=2 kV)
Ion beam developments

Production of calcium ion beam:

- Oven technique (Large capacity oven of Pantechnik)
- Filled by pure calcium
- Placed on the axis
- Temperature: 500°C - 700 °C
- Support gas: helium
- Strong getter effect

Calcium spectra, optimized for Ca^{3+}
Production of **silicon** ion beam:

- Using silane gas for plasma production
- Special care (flammable, auto ignition)
  - $1.37 \% < C < 4.5 \%$ flammable mixture
  - $C > 4.5 \%$ metastable
- System for transferring the gas from high volume high pressure (2 dm$^3$ and 50 bar) gas bottle to a smaller (50 cm$^3$ and 1.5 bar) one.
Ion beam developments

Production of silicon ion beam:
Using silane gas for plasma production

Typical silicon ion spectra. Optimized for 3+ production

CSDs of the silicon ion beam spectra as function of the optimized charge state
Technical developments

Oven
Technical developments

Oven

mw

oven
Technical developments
Technical developments
Technical developments

Sample holder

mw

oven
Technical developments

Sample holder

mw

oven
Technical developments

Sample holder

mw

oven
Technical developments

Sample holder

Place to irradiate 14 pieces samples

Place to irradiate 14 pieces samples

Place to irradiate 14 pieces samples

4-segments concentrical monitor to set the size and the homogeneity of the beam spot

5-segment profile monitor to set the x-y position of the beam
Samples were irradiated by the produced beams

<table>
<thead>
<tr>
<th>Project</th>
<th>Ion</th>
<th>$\bar{Q}$</th>
<th>Dose (ion/cm$^2$)</th>
<th>Time (hour)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>Au</td>
<td>1.3</td>
<td>$10^{16}$</td>
<td>8</td>
</tr>
<tr>
<td>B</td>
<td>Ca</td>
<td>2.2</td>
<td>$10^{17}$</td>
<td>4.5</td>
</tr>
<tr>
<td>C</td>
<td>Si</td>
<td>3.1</td>
<td>$10^{17}$</td>
<td>1.25</td>
</tr>
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First physical results

Implantation depth:

Possible to measure by SNMS (Secondary Neutral Mass Spectrometer)

Possible to predict by SRIM (Stopping and Range of Ions in Matter)
First physical results

Implantation depth:

Representative parameter to compare the calculation and measurement.

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<th>Depth (nm)</th>
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<tr>
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<td>TiO$_2$</td>
<td>2</td>
<td>1-10</td>
<td>7</td>
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<td>Ca</td>
<td>TiO$_2$</td>
<td>3</td>
<td>8</td>
<td>9</td>
</tr>
<tr>
<td>Si</td>
<td>ZrO$_2$</td>
<td>3</td>
<td>6 !</td>
<td>25</td>
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# First physical results

## Implantation depth:

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<td>Si</td>
<td>ZrO$_2$</td>
<td>3</td>
<td>6</td>
<td>25</td>
</tr>
<tr>
<td>Si</td>
<td>ZrO$_2$ +25 nm C layer</td>
<td>10</td>
<td>25</td>
<td>25</td>
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Depth profile in ZrO$_2$

30 keV Si 3+

9 keV Si 3+
Depth profile in ZrO$_2$

30 keV Si 3+

9 keV Si 3+
Depth profile in ZrO$_2$

30 keV Si 3+

9 keV Si 3+
Depth profile in ZrO$_2$

30 keV Si 3+

9 keV Si 3+
## Summary

- Production of new ion beam (Au, Ca, Si) by the Atomki ECRIS
- New irradiation facility (vacuum chamber, sample holder) to handle high number of samples
- Irradiation of the first series of samples.
- Depth profile analysis and calculation

## Plan

- Further investigation
  - Adhesion measurement
  - Morphology (AFM)
  - Clinical investigation
  - Exploring the effect of the q.
  - Improving the LCI intensities.
Acknowledgement

This work supported by the TAMOP 4.2.2.A-11/1/KONV-2012-0036 project, which is co-financed by the European Union and European Social Fund.

Thank you for your attention!