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MUSCULOSKELETAL INNOVATIONS

Development of Hard Antibacterial (TiN/Ag) Coatings on Orthopedic Instruments Fabricated from Ti-alloys

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Problems:

- TiO_2 was the main phase in the coatings.
- Ag content was high in the coating, however, the antibacterial results were not good.

Samples preparation

By changing the area ratio of Ag and Ti on the surface of the Ti target, three varied Ag content TiN-Ag coatings were obtained and named **TiN** coating, **TiN-3%Ag** coating, and **TiN-5%Ag** coating.

Parameters of Magnetron sputtering

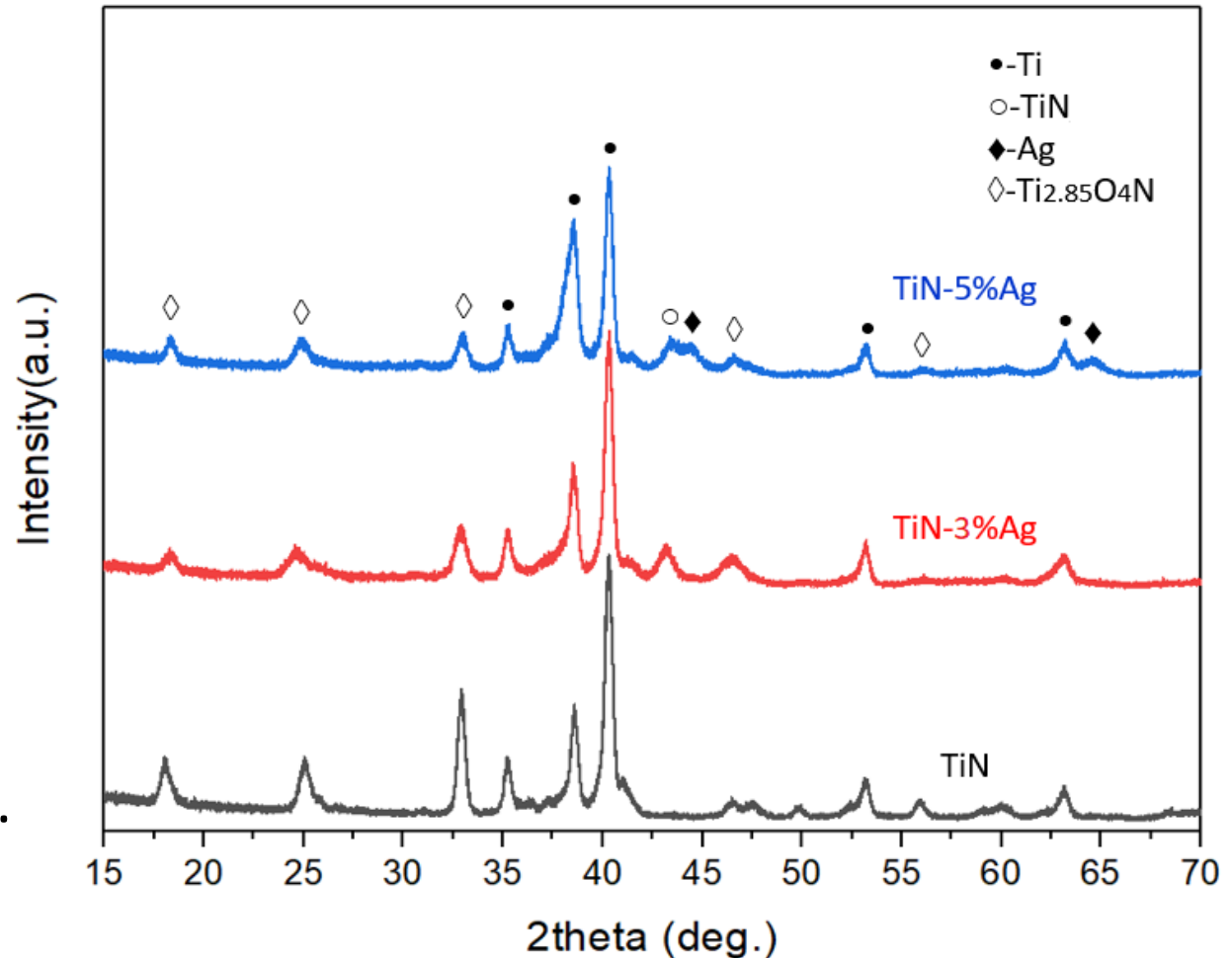
- Base pressure: 10^{-6} millitorr
- Working pressure: 35 millitorr
- N₂/Ar: 1:1
- Power density: 1.41W/cm²



XRD spectra

- **Ti_{2.85}O₄N**
(PDF# 97-017-3420)
- **TiN** (PDF# 97-023-6801)
- **Ag** (PDF# 98-000-0398)
- **Ti** (PDF# 98-001-3717)

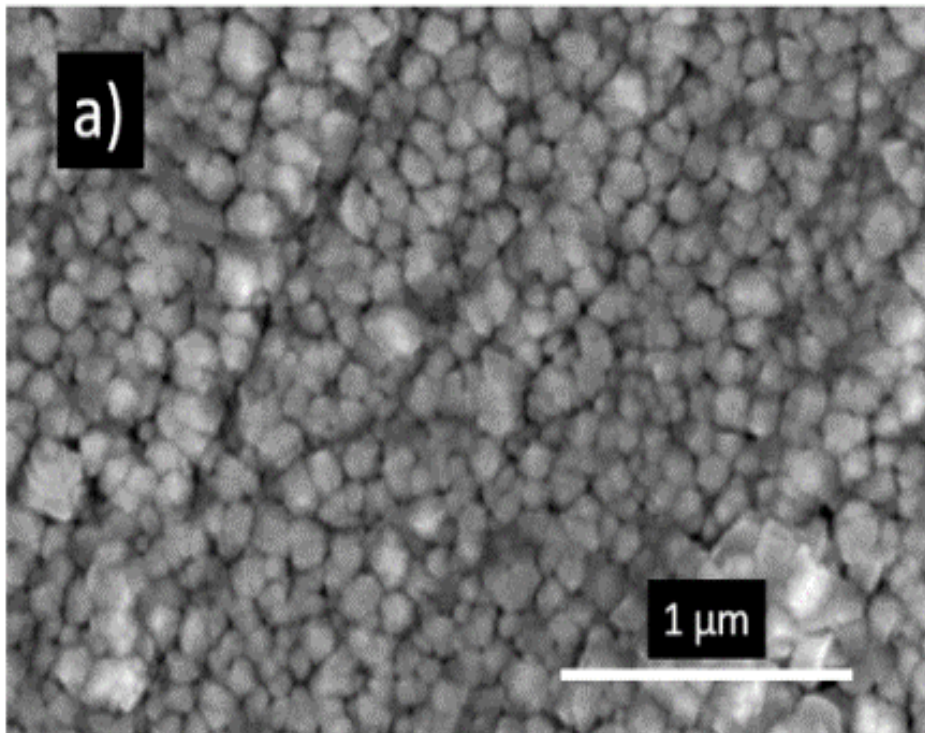
Titanium oxynitrides combines the properties of metallic oxides (optical properties) and nitrides (hardness, wear resistance).



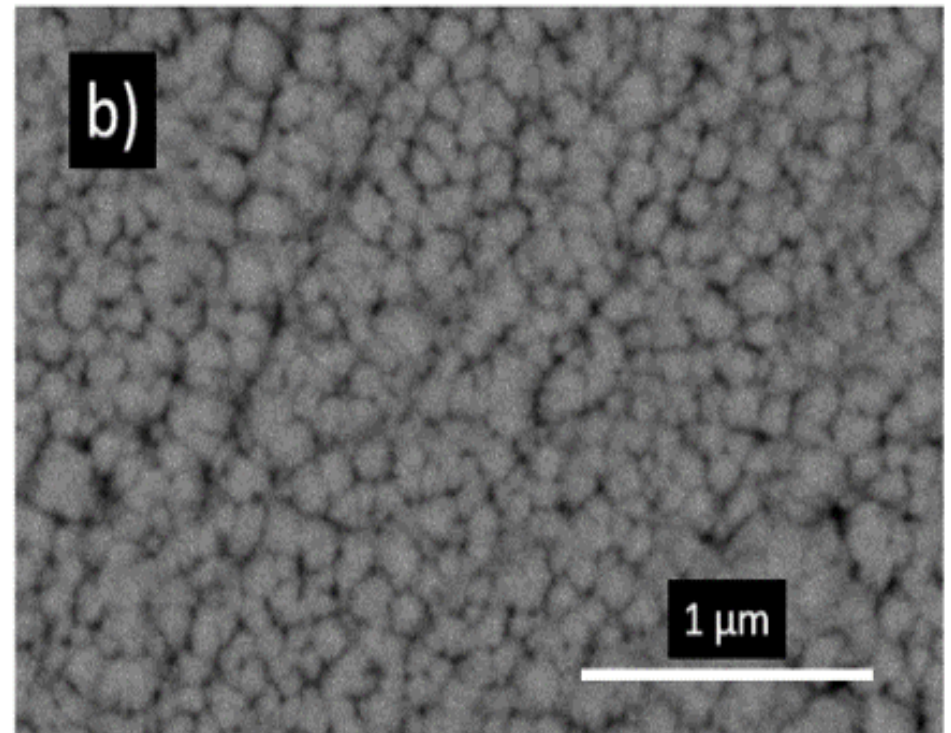
Morphological analysis

TiN coating

Second electron detector

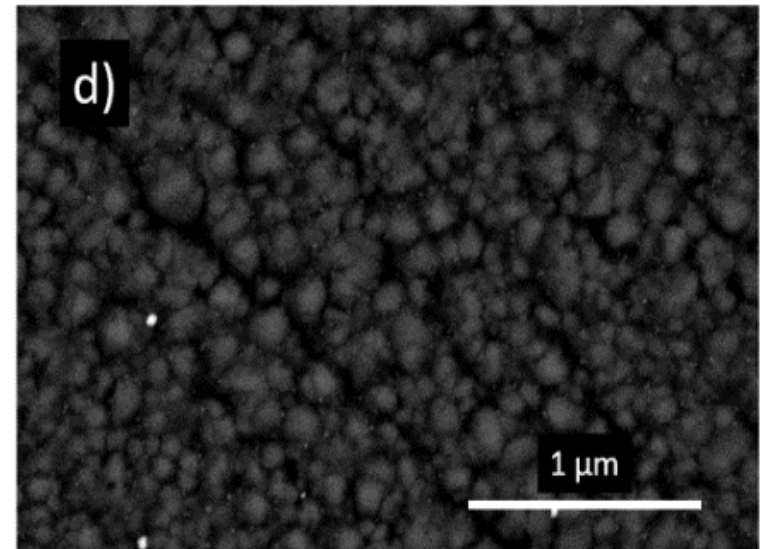
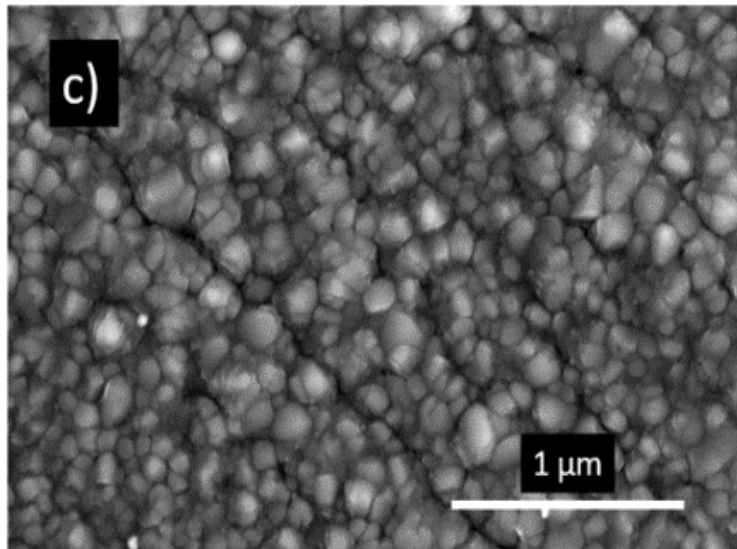


Back-scattered electron detector

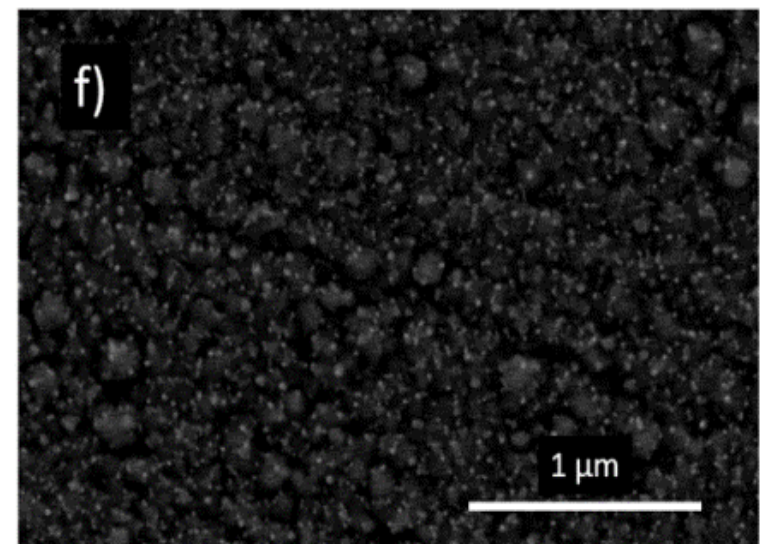
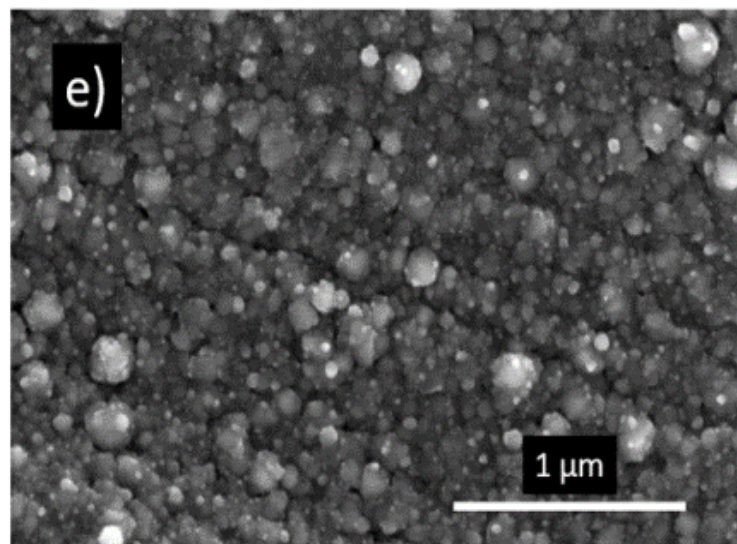


Morphological analysis

TiN-3%Ag
coating



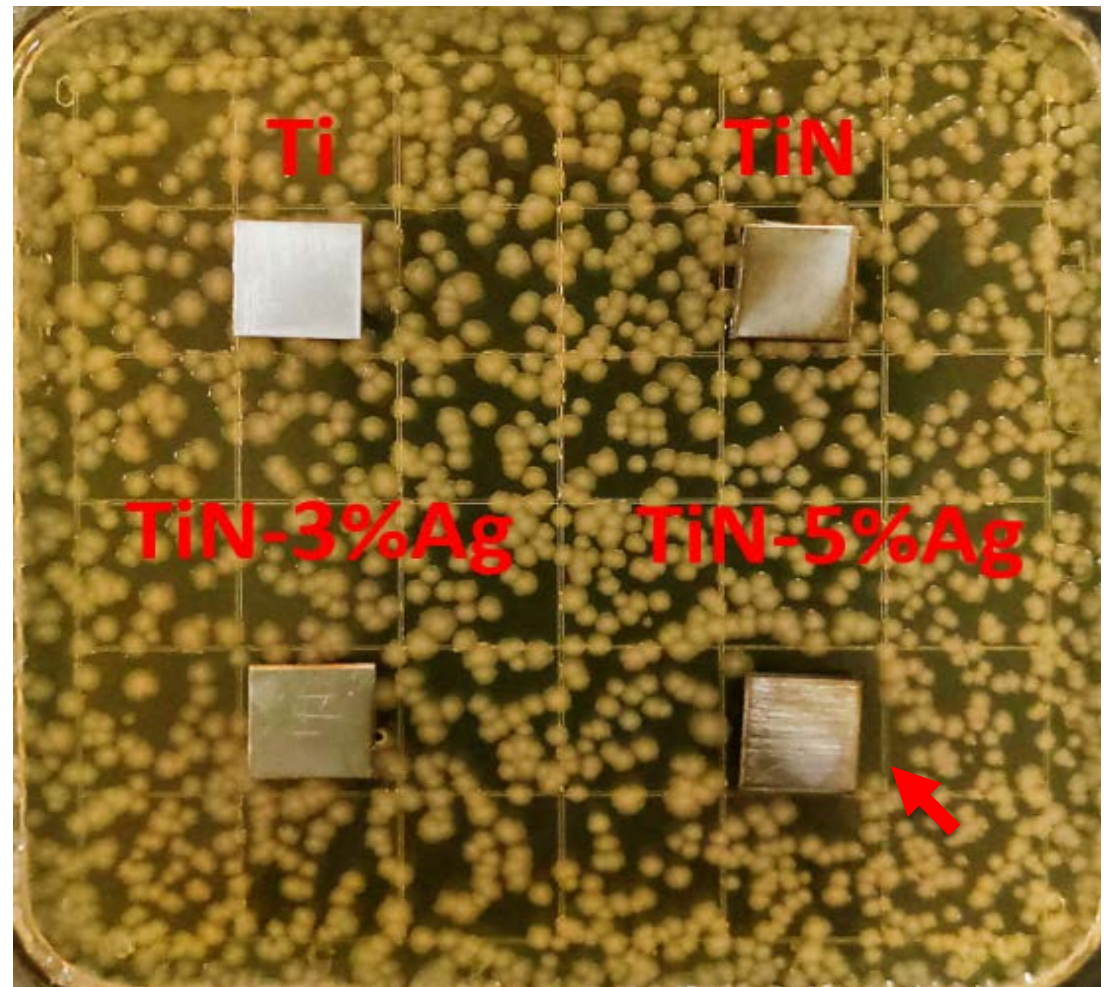
TiN-5%Ag
coating



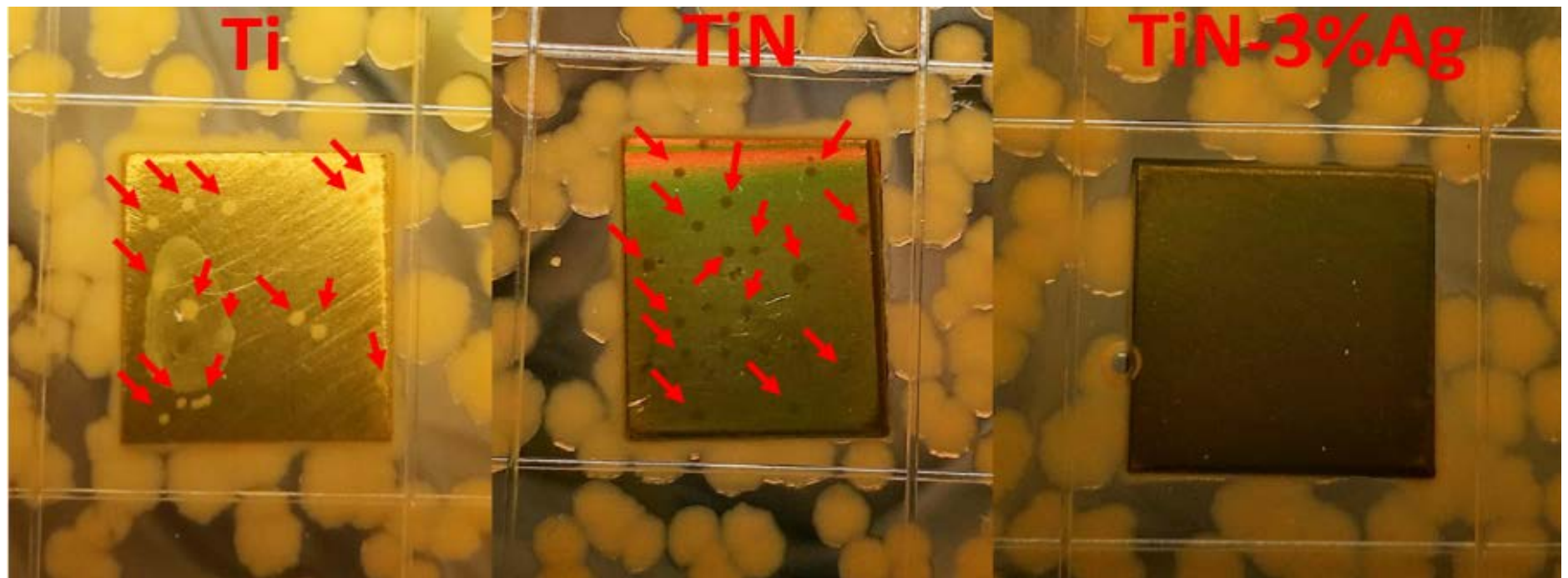
Antibacterial properties - ZOI

ZOI was found around TiN-5%Ag coating.

TiN-5%Ag coating had a strong effect of Ag-ion diffusion.

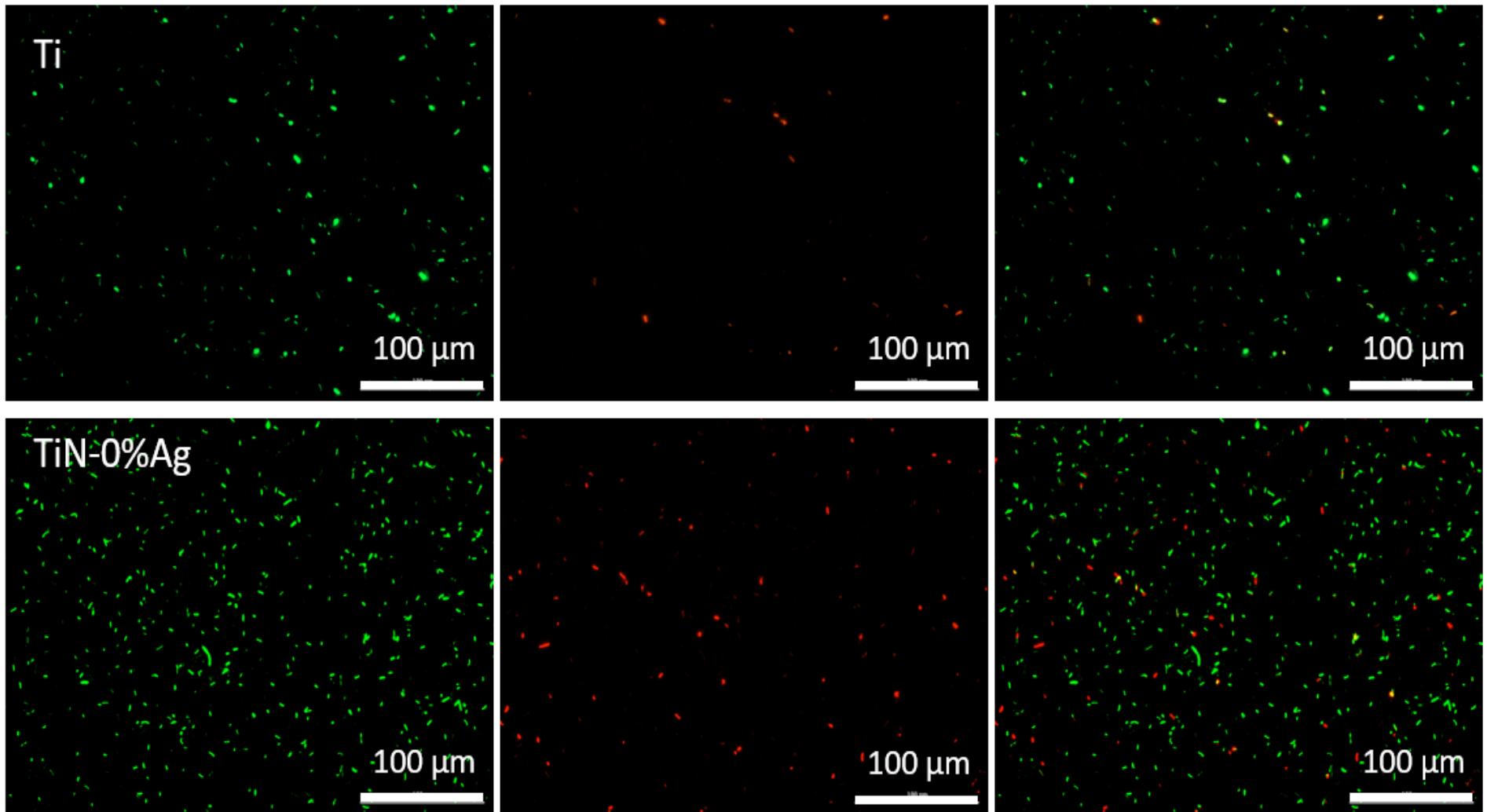


Antibacterial properties - ZOI

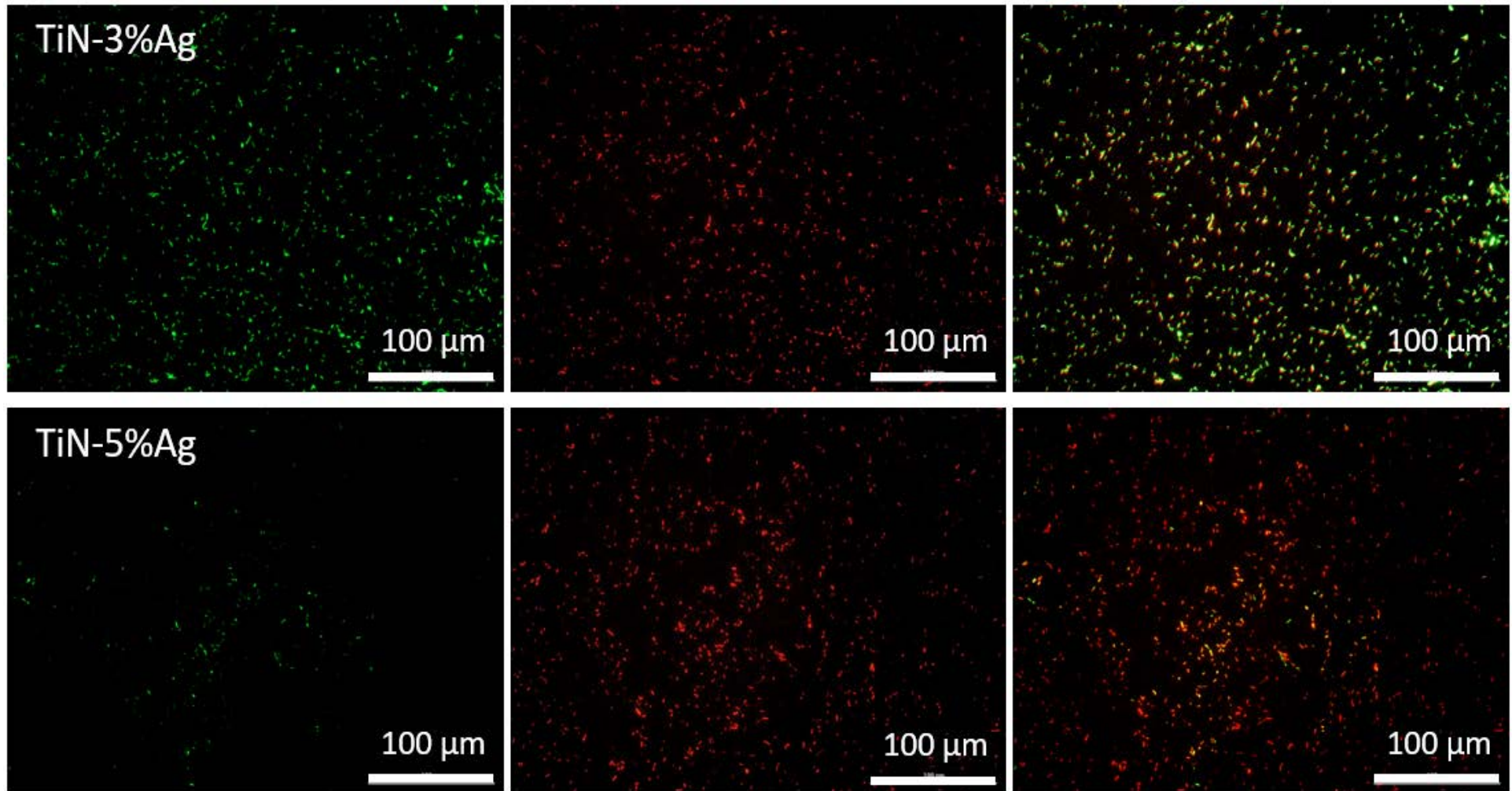


TiN-3%Ag coating was antibacterial,
but no Ag-ion diffusion was shown.

Antibacterial properties – Live/Dead Cells



Antibacterial properties – Live/Dead Cells



Antibacterial properties – Live/Dead Cells

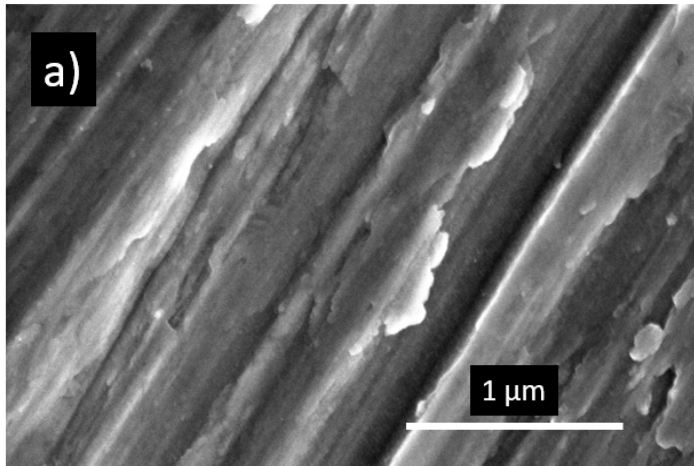
- Both of TiN-3%Ag and TiN-5%Ag coatings had contact kill properties, TiN-5%Ag was more bactericidal.

the bacteria attached on TiN coating with 5% Ag were more severely damaged, since more PI stain penetrated the bacteria cell membranes and took the place of syto9, reducing the green fluorescence and showing more red fluorescence.

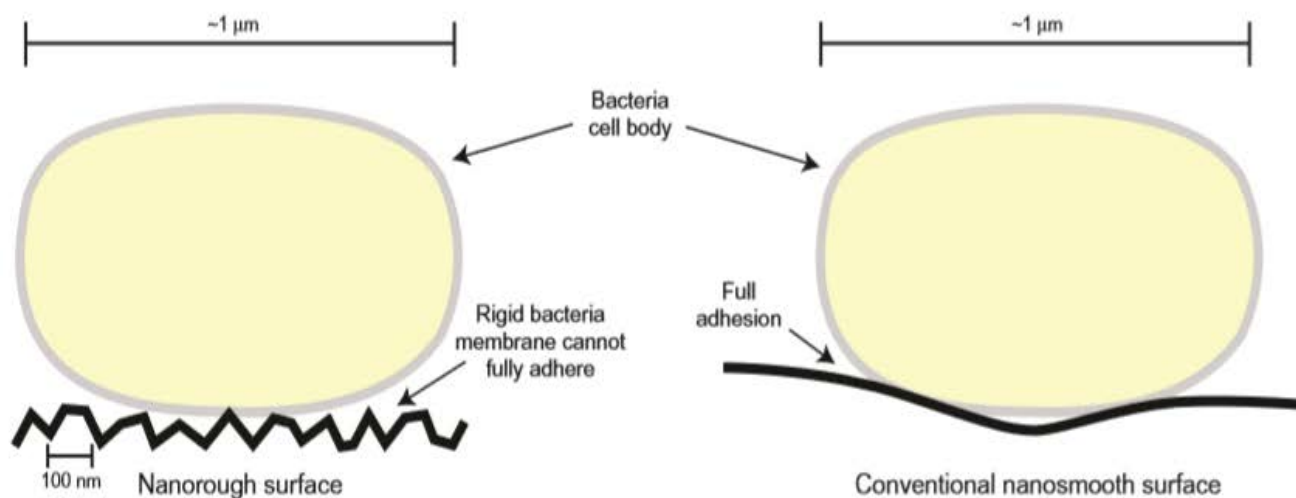
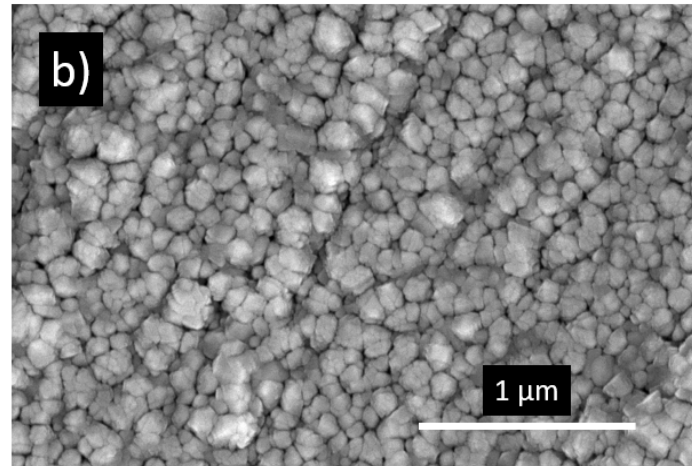
Antibacterial properties – Live/Dead Cells

- Ti block had the least bacteria attachment

Ti block



TiN coating

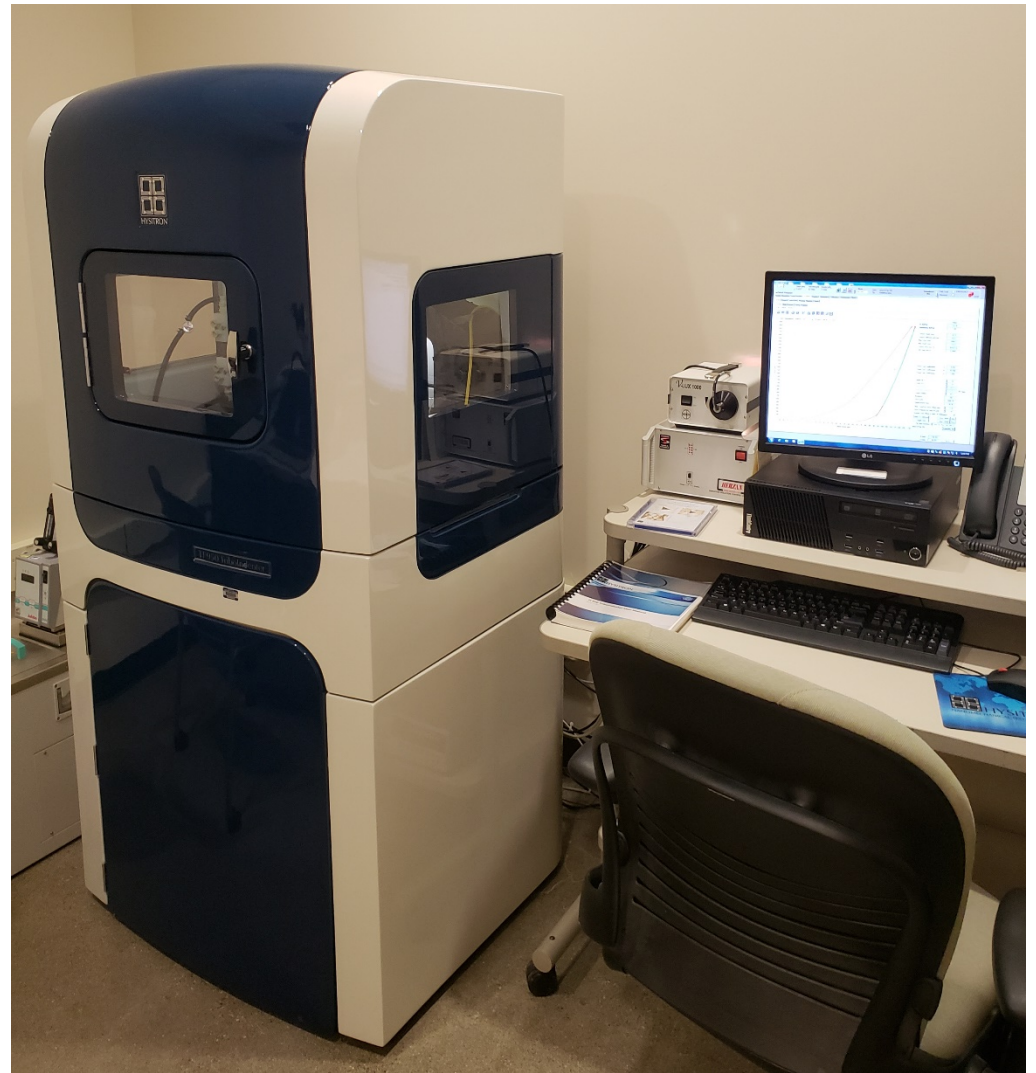


Hardness measurement

- Hysitron Ti-950 TriboIndenter
- TriboScan

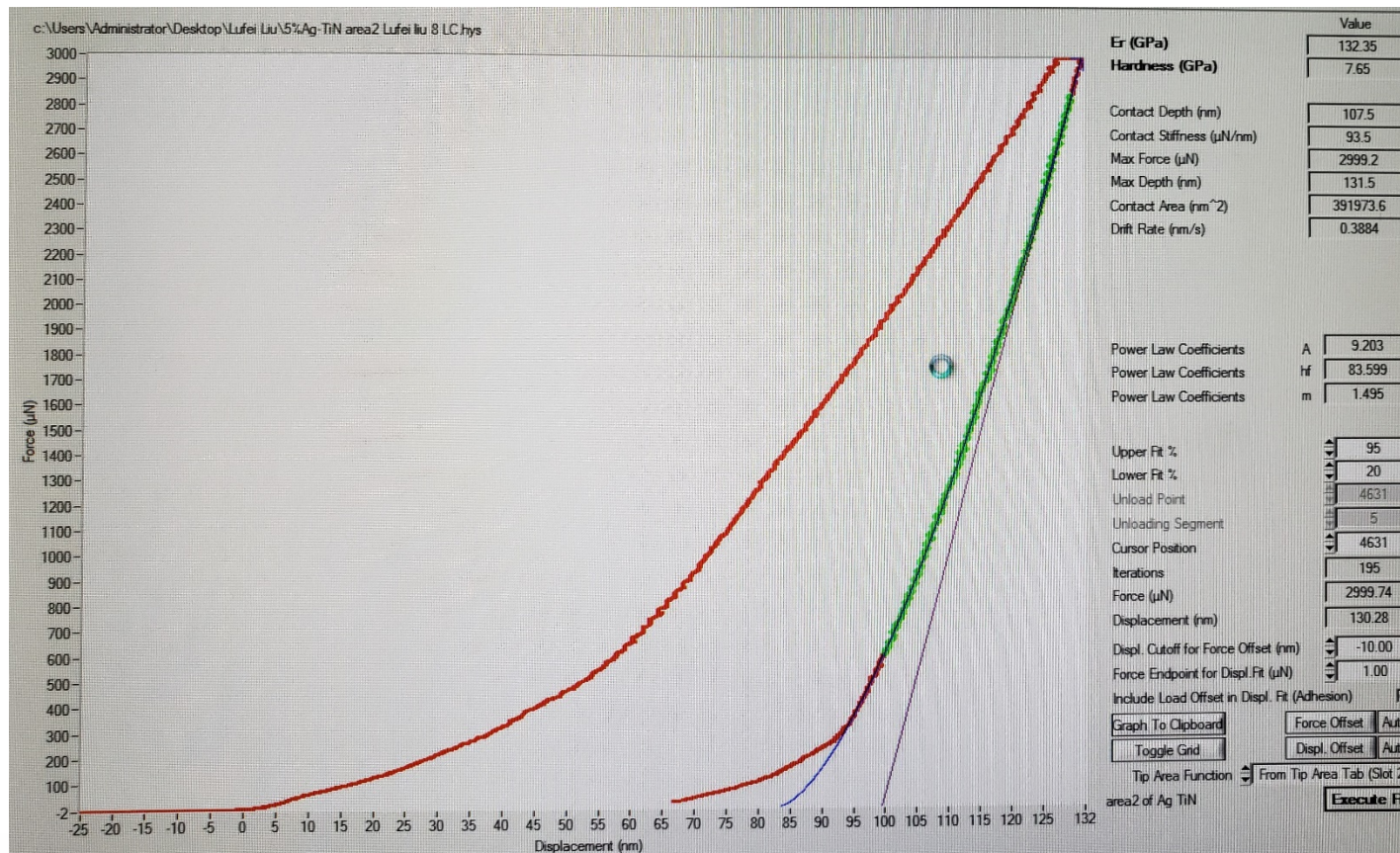
load: 3000 μ N
holding time: 10 s.

The portion from 20% lower part to 95% upper part of the unload curve was used for calculation



Hardness measurement

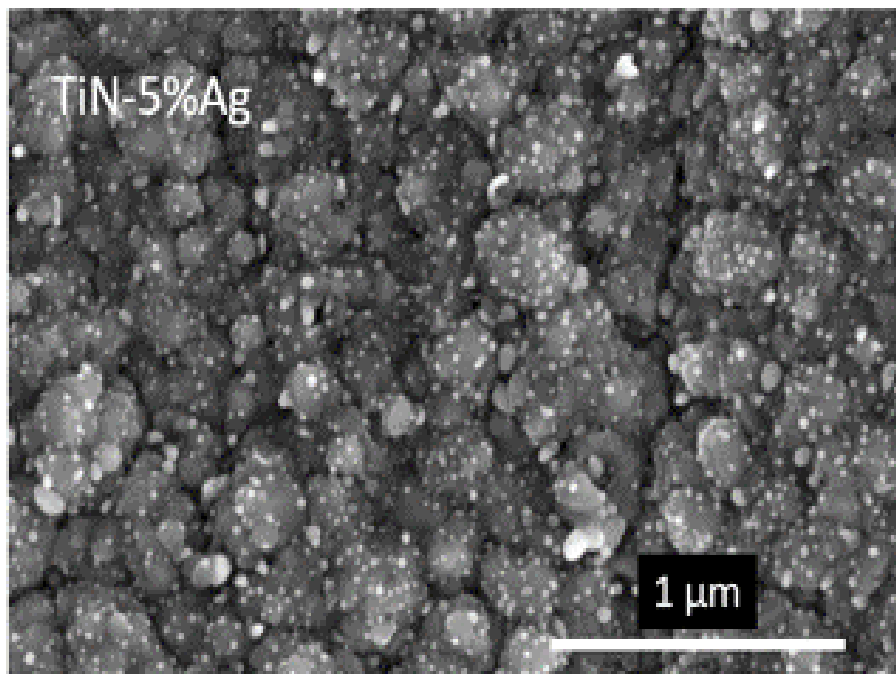
- Hardness of TiN-5%Ag coating: 7.03 ± 0.48 GPa
- Ti-6Al-4V: 3.3 GPa



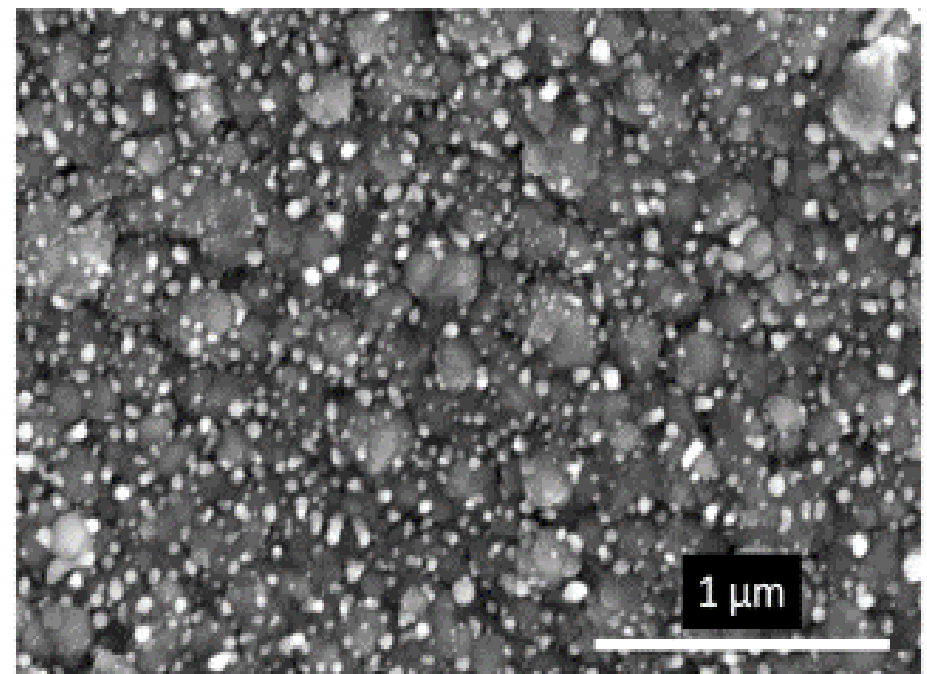
Impact of autoclaving cycles

More Ag nanoparticles were presented and became larger on the surface of the coating after autoclaving treatments.

1 Autoclaving cycle



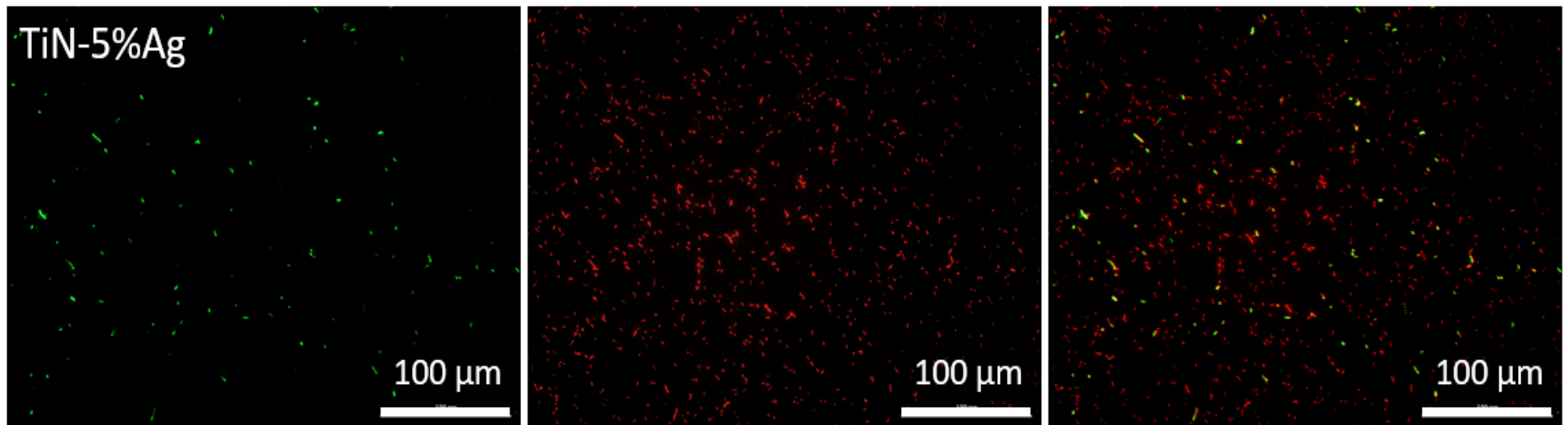
5 Autoclaving cycles



Impact of autoclaving cycles

No impact on antibacterial property was shown

After 5 Autoclaving cycles



Conclusions

- **Compositions:** TiN, Ti_{2.85}O₄N, and Ag. Coating particles are highly crystallized and in a dense arrangement, Ag nanoparticles with a diameter around 30nm distributed uniformly across the coating surface.
- **Antibacterial Properties:** Both TiN-3%Ag and TiN-5%Ag had antibacterial properties, TiN-5%Ag was more bactericidal and had a Ag-ion diffusion effect.
- **Hardness:** 7.03±0.48 GPa
- **Impacts of Autoclaving treatment:** Ag nanoparticles on the surface had an aggregation effect, no influence was shown on antibacterial properties.

- **Future works:**

More works are needed in the coating process to reduce the oxygen content. **Nitrogen-rich film is better for the mechanical and biocompatible performances.**

- **Timeline:**

Finish project by September 2018;
Finish final reports by October 2018.

Thanks!