



## Hard nanostructured coatings on plasma nitrided Ti6Al4V

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## Overview

- Problem of coating spallation from substrate after first erosion tests
- Improving coating adhesion to Ti6Al4V by two types of plasma nitriding approaches:
  - RF nitriding
  - HiPIMS nitriding
- Nanocomposite TiSiN and multilayer TiN/Ti coating on HiPIMS nitrided surface
- Water erosion tests of nitrided samples and coated samples with HiPIMS nitrided surface
- Summary and outlook

## **Coating spallation under water erosion**

#### **TiN-TiSiN multilayer**



#### **Ti-TiSiN** multilayer



Severe spallation of coating after 30 seconds of erosion test! Problem of coating adhesion to Ti6Al4V!

> Erosion time: **30** s Nozzle: **400 μm** Impinging speed: **350 m/s** Estimated droplets size: **450 μm** Water pressure: **30 psi** Water flow: **0.05 l/min**

## Problem of depositing hard coating on soft substrate



- soft substrate does not offer good support for brittle coating under high loads
- large difference in elastic modulus between coating and substrate causes high stress on the coating/substrate interface causing poor adhesion and failure during tribological operation
- difference in crystal structure of coating and Ti6Al4V is not favorable for adhesion



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## Improving adhesion of coating to Ti6Al4V

**Duplex process: plasma nitriding + hard coating** 



- nitrided surface improves load-bearing capacity
- nitriding provides more gradual stress distribution from substrate to the coating due to smaller mismatch in shear modulus
- better lattice match and chemical affinity between nitrided surface and nitride coatings improves adhesion
- when coating is removed nitriding prevents fast erosion of substrate

plasma nitriding

Molinari, G. Straffelini, B. Tesi, T. Bacci, and G. Pradelli, *Wear* **203–204**, 447 (1997) M. K. Lei, Z. L. Zhang, and T. C. Ma, *Surf Coat Technol* **131**, 317 (2000) D. Nolan, S. W. Huang, V. Leskovsek, and S. Braun, *Surf Coat Technol* **200**, 5698 (2006) F. Yildiz, A. F. Yetim, A. Alsaran, and A. Çelik, *Surf Coat Technol* **202**, 2471 (2008)

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### **Approaches to plasma nitriding of Ti6Al4V**



### Nitrided samples by RF and HiPIMS

#### **RF** nitriding

**HiPIMS** "nitriding"



| type of<br>nitriding | temp<br>(°C) | p<br>(mtorr) | φ <sub>Ar</sub><br>(sccm) | φ <sub>N2</sub><br>(sccm) | time<br>(h) | voltage<br>(V) | thickness<br>(μm) | H (0.5 N)<br>(GPa) |
|----------------------|--------------|--------------|---------------------------|---------------------------|-------------|----------------|-------------------|--------------------|
| RF                   | 700          | 700          | 10                        | 90                        | 4           | -1000          | ~30               | 8-10               |
| HiPIMS               | 450          | 5            | 35                        | 2                         | 3           | -800           | ~2-3              | 6-8                |

### Deposition of hard coatings on nitrided Ti6Al4V

#### nanocomposite coating

#### TiSiN

| HiPIMS nitrided | \$~2 μm<br>\$~3 μm |
|-----------------|--------------------|
| Ti6Al4V         |                    |

#### multilayer coating

#### 3×(TiN/Ti)/TiN – 10 layers





| process                           | TiSiN | TiN/Ti |
|-----------------------------------|-------|--------|
| # of layers                       | 1     | 10     |
| nitriding time (h)                | 2     | 2      |
| deposition time (h)               | 4     | 3.5    |
| deposition temp (°C)              | 450   | 450    |
| p (mtorr)                         | 5     | 5      |
| φ <sub>Ar</sub> : φ <sub>N2</sub> | 17:1  | 17:1   |
| substrate bias (V)                | -45   | -45    |
| total thickness (µm)              | ~5    | ~5     |
| hardness                          | 35    | 22     |
| elastic modulus                   | 240   | 190    |
|                                   |       |        |



### Water erosion test of nitrided samples

#### **RF** nitriding



#### **HiPIMS** "nitriding"







Erosion time: **2 min** (30s + 30s + 60s) Nozzle: **400 μm** Impinging speed: **350 m/s** Droplets size: **~450 μm** Water pressure: **30 psi** Water flow: **0.05 l/min** 





speed: 350 m/s, nozzle: 400 μm, droplets size: 450 μm, water pressure: 30 psi, water flow: 0.05 l/min

### **Erosion track of coated samples**

#### **TiSiN//HiPIMS nitrided**



#### TiN/Ti//HiPIMS nitrided





## Comparison of erosion tracks on coated and nitrided samples after 2 minutes test

#### **HiPIMS nitrided**



#### **HiPIMS nitrided**



#### **TiSiN//HiPIMS nitrided**



#### **TiN/Ti//HiPIMS nitrided**



## Summary

- **plasma nitriding** of Ti6Al4V was used to improve adhesion and load-bearing capacity for hard coatings
- two approaches for plasma nitriding were chosen:
  **RF and HiPIMS nitriding**
- **TiSiN nanocomposite** and **TiN/Ti multilayer** coatings were deposited over HiPIMS nitrided substrate
- nitriding alone improves water erosion resistance
- coated samples with HiPIMS nitrided layer show better water erosion resistance compered to HiPIMS nitrided samples

## Plan of activities until the end of year

- mechanical, structural and chemical analysis of nitrided layer and coatings
- continuation of improving plasma nitriding: **DC nitriding**
- deposition of thick TiSiN nanocomposite and TiN/Ti multilayer coating (~10 μm) on RF or DC and HiPIMS nitrided surface
- incorporation of **carbon** into TiSiN coating for increasing  $H^3/E^2$  ratio and deposition of thick coating (~10 µm) on nitrided surface

| Research Activity               | Progress    | Risk of going beyond 2013 |
|---------------------------------|-------------|---------------------------|
| Literature Review               | finished    | no                        |
| Materials Selection             | finished    | no                        |
| Process Development             | finished    | no                        |
| <b>Coating Characterization</b> | in progress | no                        |
| Testing and optimization        | in progress | no                        |