Effects of Plasma Enhanced Atomic Layer Deposition Processing Parameters on the Friction and Wear of Multi-metal Nitride Films.

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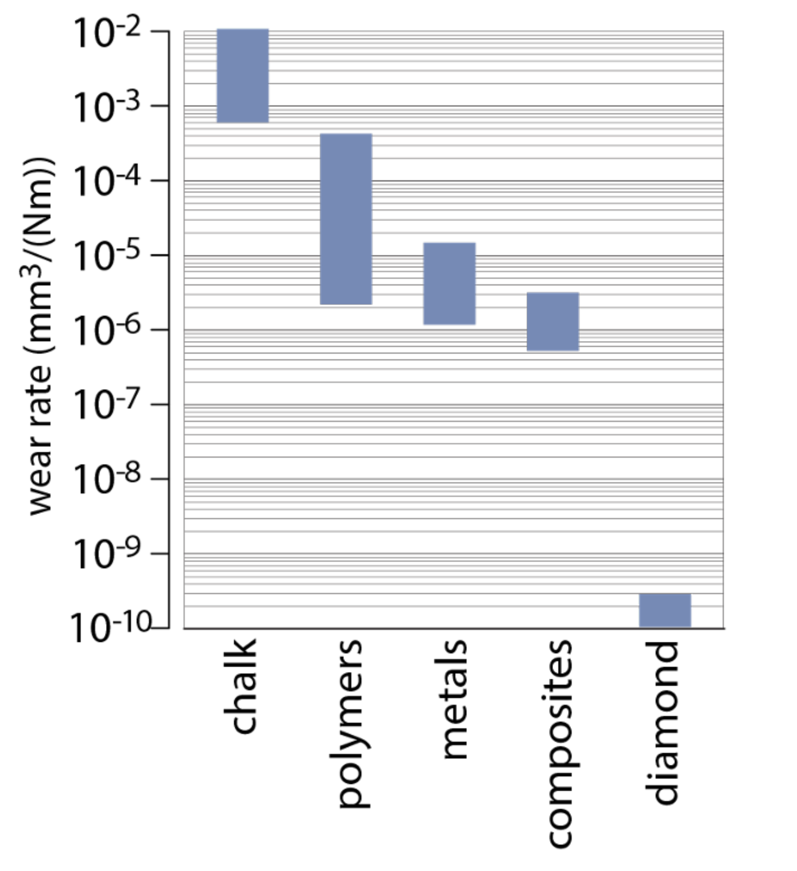
Introduction

- Atomic Layer Deposition is technique used to deposit thin-films by exposing a material (substrate) to different precursors that react in a self-limiting way with a heated substrate.
- Plasma Enhanced Atomic Layer Deposition (PEALD) uses plasma to help the substrate react with the chemicals at a lower temperature.
- Nitride coatings, such as TiVN or TiMoN, have good wear resistance, low friction, and low electrical resistance. Both nitrides exhibit self-lubricating behavior when sliding in humid conditions. A thin layer of lubricious oxide (MoO_x and V_xO_y) forms on the surface reducing the coefficient of friction [1], [2].
- The materials that are being studied can be used in industries such as microelectronics, MEMS, NEMS, and biomedical implants. The low wear and friction make TiVN or TiMoN suitable replacements for conventional materials.



- Preliminary experiments have found that the wear rates for TiVN are ~10x lower than wear rates for DLC coatings [3].
- This project aims to study the mechanical properties of PEALD nitride coatings, specifically TiVN and TiMoN, and to establish process-structure-property relationships.
- Process changes, such as substrate cleaning procedures can impact the coating microstructure and tribological properties.
- Three different substrate cleaning procedures were performed prior to deposition: Organic solvents, Organic solvents + Standard Clean 1 (SC1), and Organic solvents + Ozone (O_3).

MEMS
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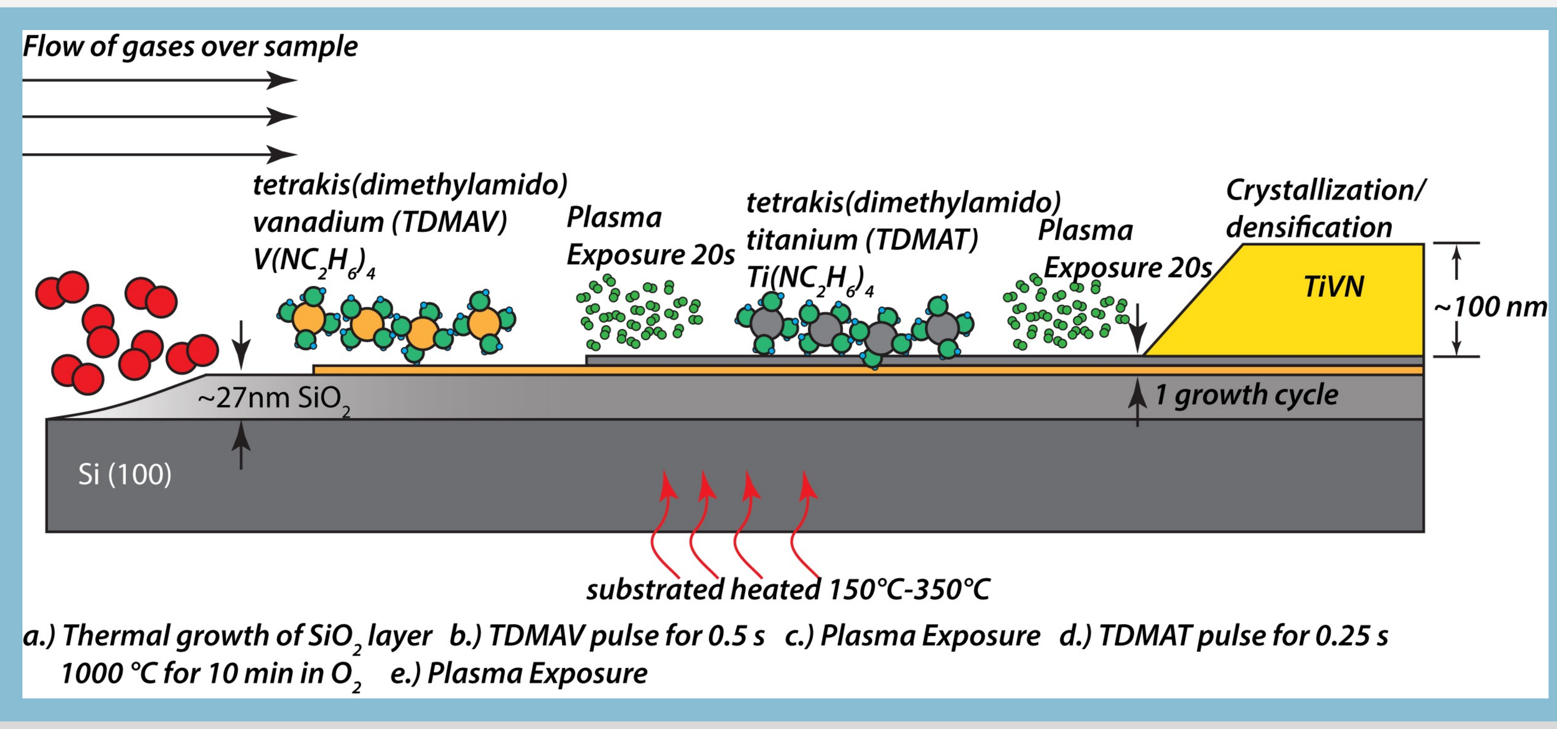

Methods

Experiment Parameters:

Friction and wear test were performed on the nitrides to study how the different preparation methods affected their tribological (mechanical) properties. The coatings were tested on a linear bidirectional reciprocating tribometer with a ball-on-flat configuration shown in fig 1(a). The velocity and track length was 1 mm/s and 1 mm respectively for all tests.

Sample	Humidity (RH)	Load (N)	Counter Surface	Cycles
TiMoN	60%	0.1	3 mm sapphire ruby ball	10000
TiMoN	60%	0.1	3 mm sapphire ruby ball	30000
TiMoN	60%	0.1	3 mm sapphire ruby ball	60000
TiMoN	60%	0.1	3 mm sapphire ruby ball	100000
TiVN	50%	0.1	3 mm sapphire ruby ball	6000
TiVN	50%	0.1	3 mm sapphire ruby ball	11000

Methods

Data Collection:

For the TiMoN coatings, surface scans were obtained using an optical profilometer after the stripe tests. For the TiVN coatings, the optical profilometer was also used to obtain the surface scans, but for this sample the scans were taken as the test progressed since it was done on the *in situ* tribometer. The profilometry data was used to determine the cross-sectional area of the wear scar to calculate the wear rate as shown in Fig 1 (c). The formula used to calculate the wear rate (K) is the following:

K = wear rate
V = volume lost
F_N = normal force
d = sliding distance
A = cross sectional area
s = track length

$$K\left(\frac{mm^3}{N\cdot m}\right)=\frac{V(mm^3)}{F_N(N)\cdot d(m)}=\frac{1000\left(\frac{mm}{m}\right)\cdot A(mm^2)\cdot s(m)}{2F_N(N)\cdot C(cycles)\cdot s(\frac{m}{cycle})}=\frac{1000\cdot A}{2F_N\cdot C}$$

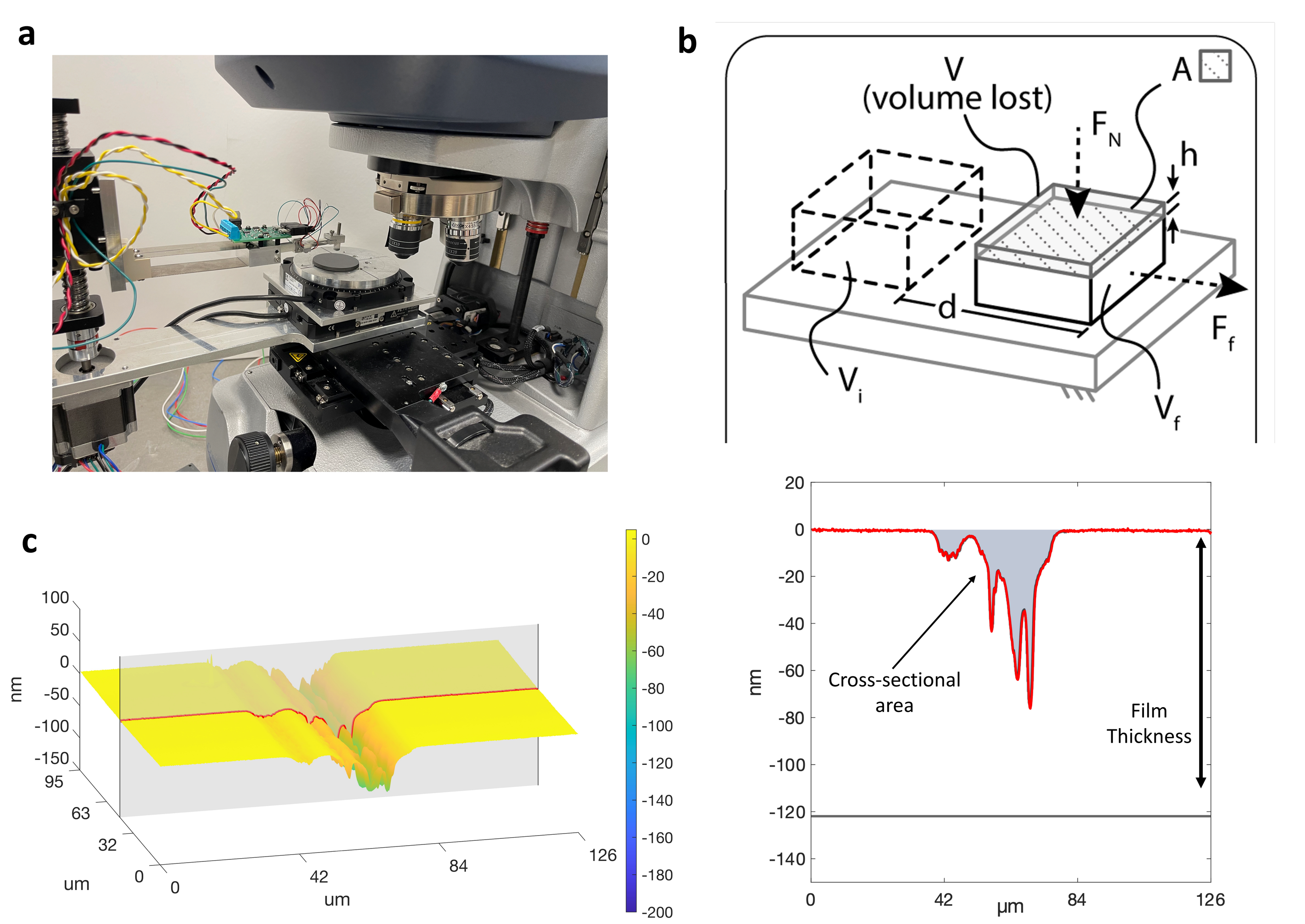

Fig. 1. (a) Tribometer (b) Wear rate – volume loss representation. (c) 3D wear track and cross-sectional area.

Results

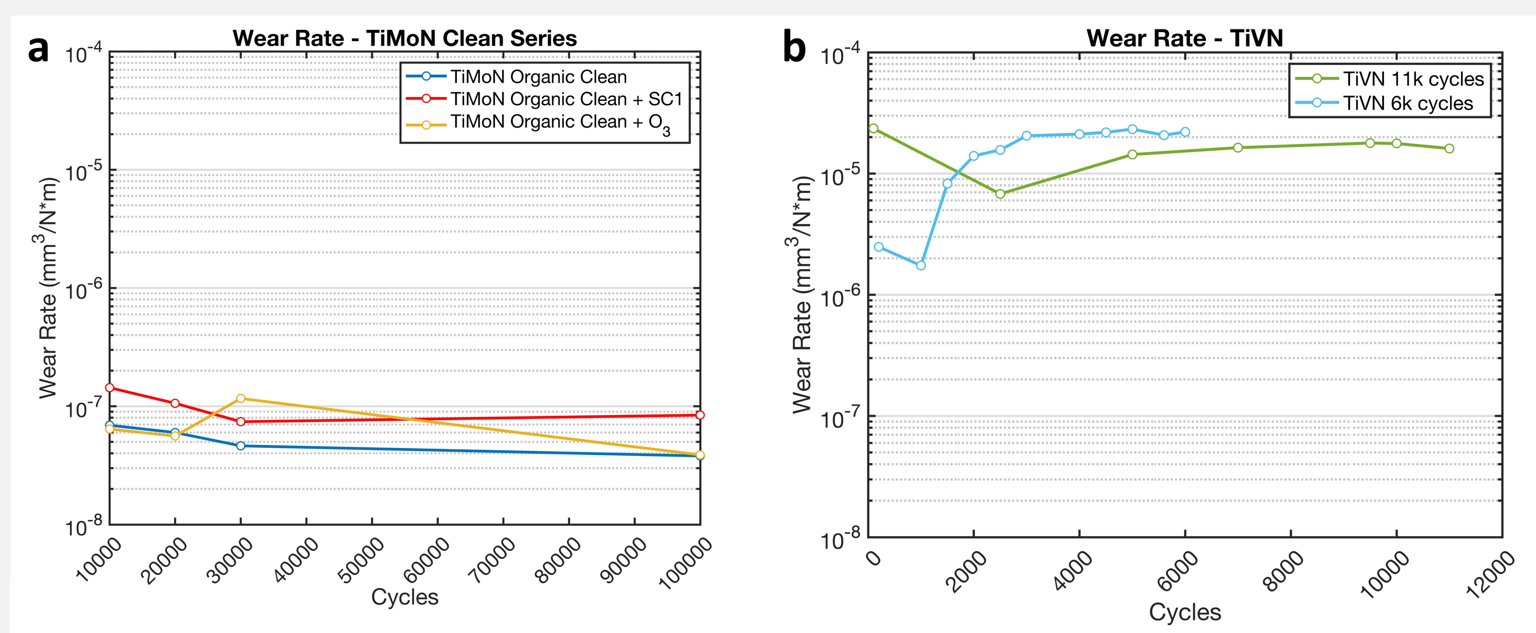

Fig. 2. (a) TiMoN Clean series Wear Rate (b) TiVN Wear Rate

Figure 2(a) shows the measured wear rate for TiMoN coating as a function of cycles. Delamination was observed in the TiMoN films with SC1 and O_3. The TiMoN organic clean had the lowest wear in the clean series with a wear rate of $\approx 3.8\times10^{-8}\,\frac{mm^3}{N\cdot m}$.

Figure 2(b) shows the wear rate for TiVN coatings as a function of cycles. Despite having a high wear rate, a crack propagation behavior was observed outside the wear track (see fig. 3a,b). This cracking might be due to a high residual stress on the PEALD films. The cracks progressively increased and propagated as the film worn, as shown in figure 4 for the 6000-cycle test.

Results

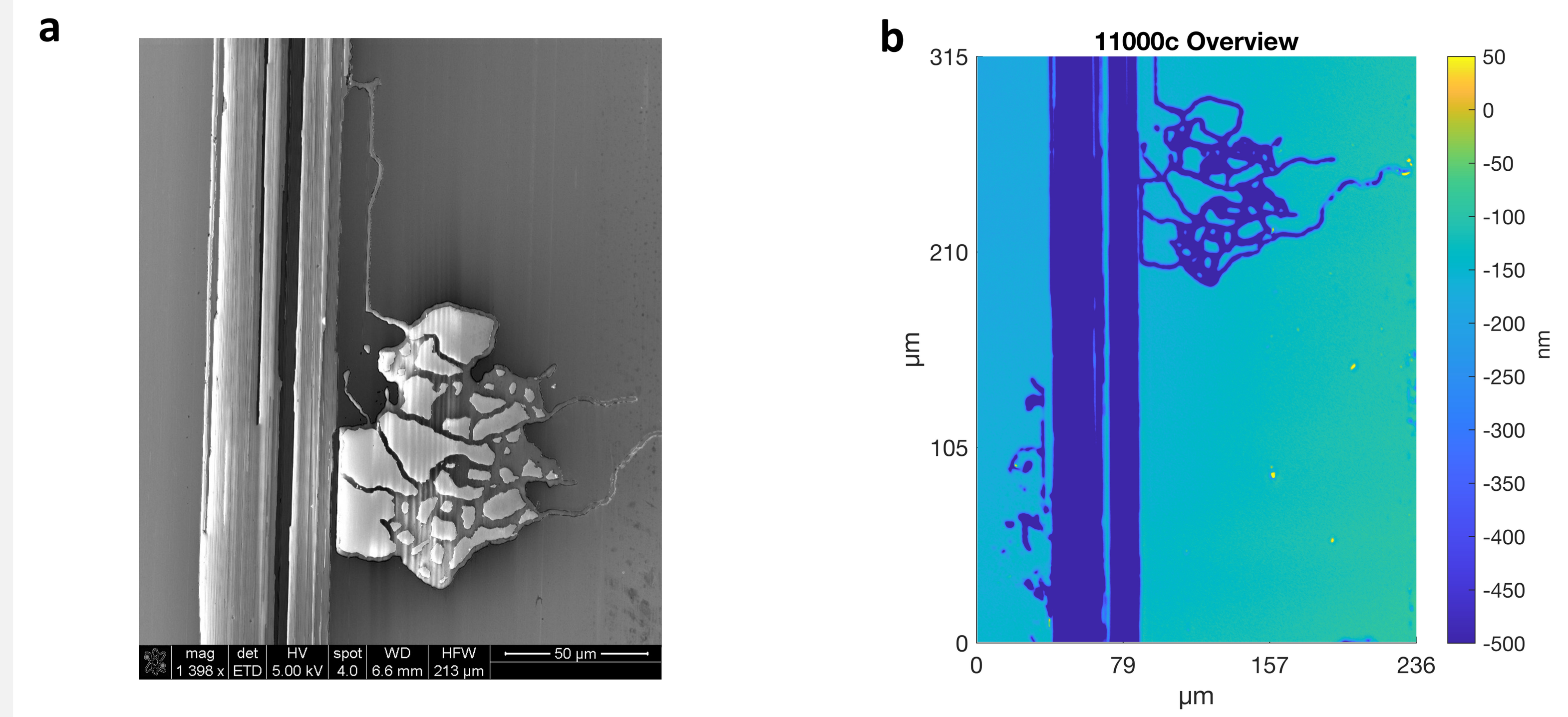

Fig. 3. 11000 cycle TiVN wear track scans (a) Scanning electron microscopy (SEM) (b) Scanning White Light Interferometry (SWLI)

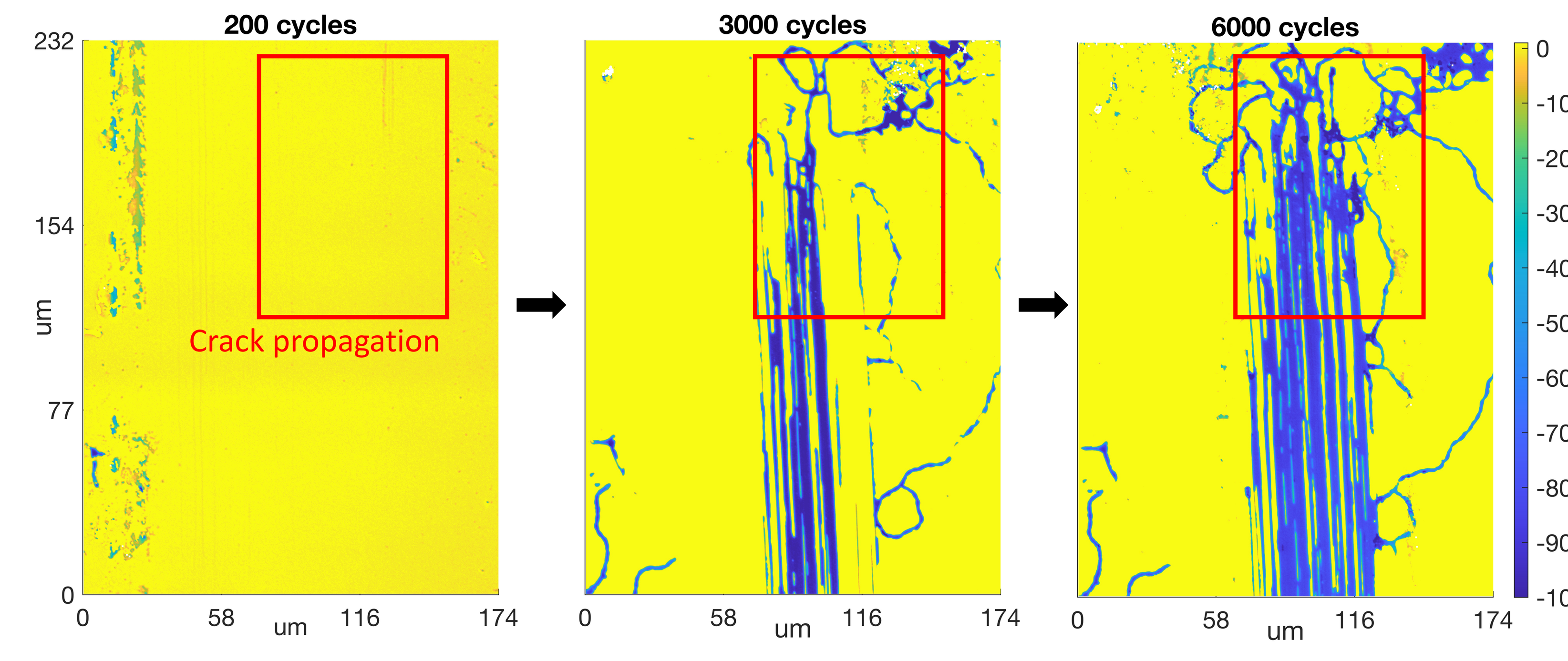

Fig. 4. Evolution of TiVN sample – Crack propagation

Discussion

- Tribology is the study of interacting surfaces in relative motion, taking into account friction, wear, and chemical mechanisms that occur during the surface interactions.
- PEALD makes possible the growth of really thin layers (~120 nm) that can be wear resistant or fail, depending on the interface conditions.
- Process-structure-property relationships will determine how well an interface will perform.
- TiMoN coatings had ultra low wear rates and are an example of good interfaces. They are an example of good PEALD coatings with low friction and wear rates.
- The particular TiVN coating tested is an example of a coating where the interface fails, in this particular case it failed with a cracking behavior.
- Residual stress in the coating is a determining factor on the wear rate of the PEALD coatings. The film needs certain amount of residual stress to have a low wear rate. However, if the residual stress in the coating is too high, the interface will fail.
- Future work can be directed to heat treatments to change residual stresses in PEALD coatings and achieve a good balance between residual stress and wear rate.

References

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