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Materials Science and Engineering
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Plasma nitriding plus oxidizing as a protective treatment for AISI 4140 steel

Eugenia L. Dalibón¹, Pablo Vergniaud¹, Esteban Karges², Amado Cabo², Sonia P. Brühl^{1,3}

Universidad Tecnológica Nacional – UTN, Concepción del Uruguay, Argentina

IONAR S.A, Buenos Aires, Argentina

National Council for Research and Technology, CONICET; Argentina

Motivation

Steels

Nitrided layer

Hardness and good wear resistance, sometimes poor corrosion resistance

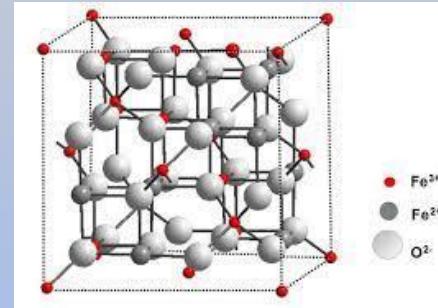
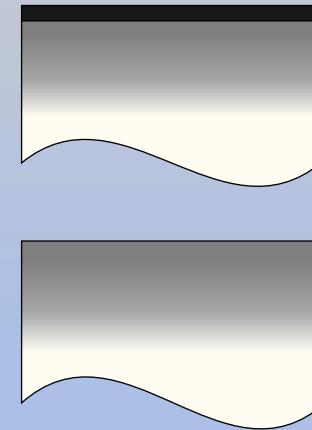


Oxide layer

Good corrosion resistance and also low friction coefficient

AISI 4140
steel

Nitrided layer + Oxide
Nitrided layer



Magnetite



Main Goals

1. To study of the microstructure and surface properties of the nitrided and oxidized layer.
2. To evaluate the wear behaviour under different conditions such as rotational- reciprocating sliding, and fretting.
3. To analyze the corrosion resistance using Salt Spray Fog Test and potentiodynamic polarization.



Experimental

1. Samples: Heat treated AISI 4140 steel discs

Fe (%)	C (%)	Si (%)	Mn (%)	Mo (%)	Cu(%)	Cr (%)
96.9	0.398	0.304	0.789	0.23	0.142	1.05

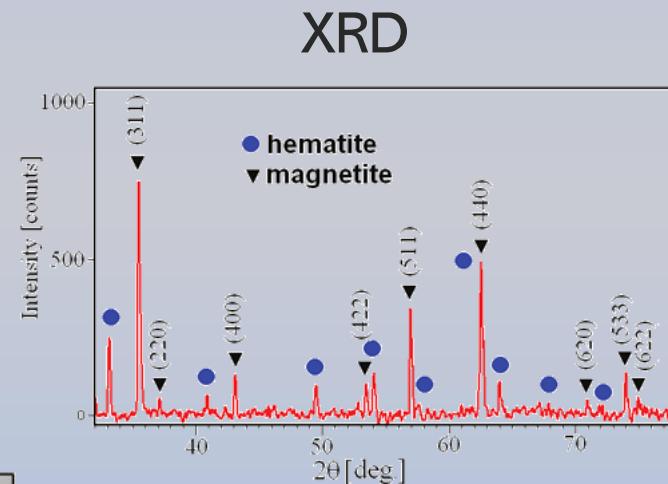
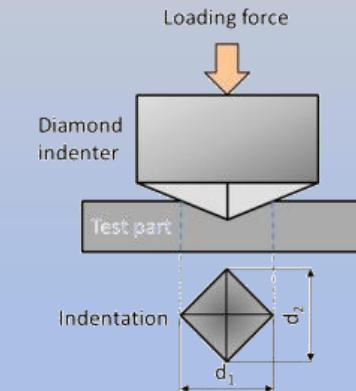
2. Nitriding: IONAR S.A. (Arg), DC pulsed plasma nitriding

20% N₂ – H₂, 500 °C, 15 h

3. Oxidizing: water steam, 400 °C, 1 h

4. Characterization:

Optical Microscopy, SEM, XRD



Vickers Hardness

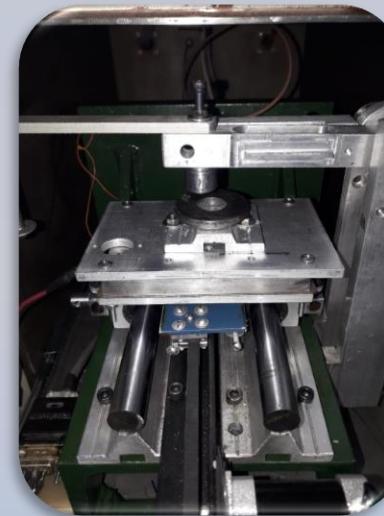
Experimental

Wear

Adhesive
Pin-on-Disk
ASTM G99
3 N, 500 m

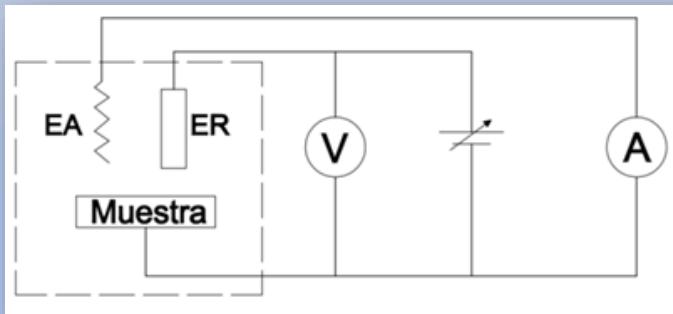


Reciprocating
sliding tests
5 mm length
354 Mpa
PAO 6 Oil



Corrosion

Potentiodynamic polarization / NaCl



Salt Spray
Fog tests
following
ASTM B117
100 h

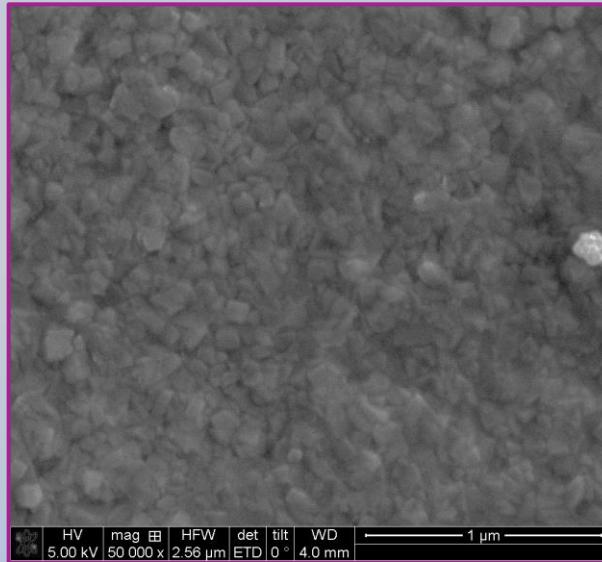


Fretting tests
A: 80 µm
Load 2.1 N
17 Hz, 15 min

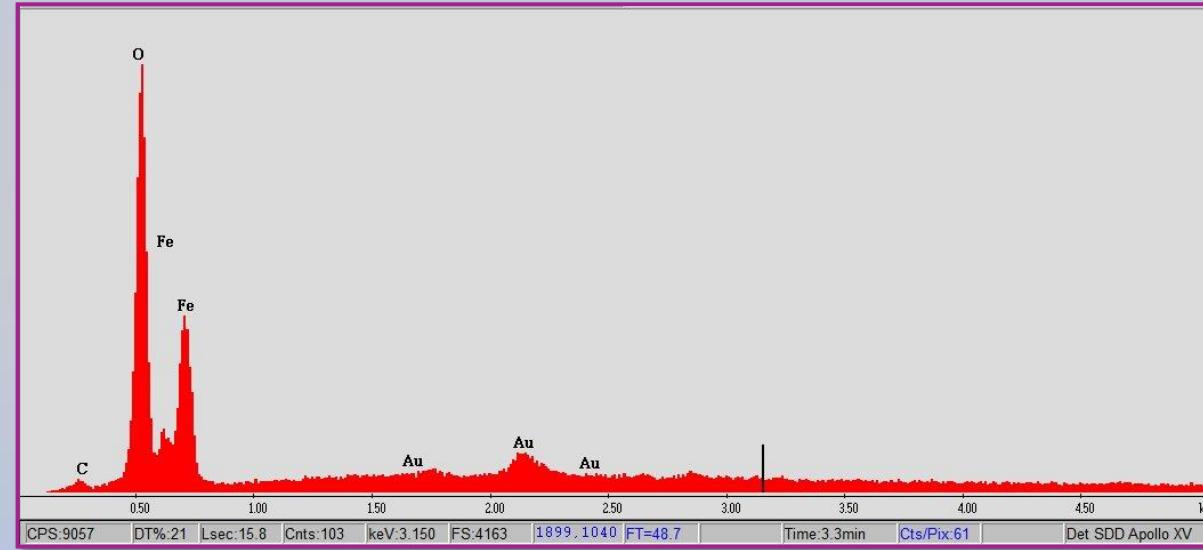


Results

Composition



SEM image on the surface



EDS measurement

Sample	% At. O	% At. Fe
Nitrided and oxidized steel	38,8	56,8



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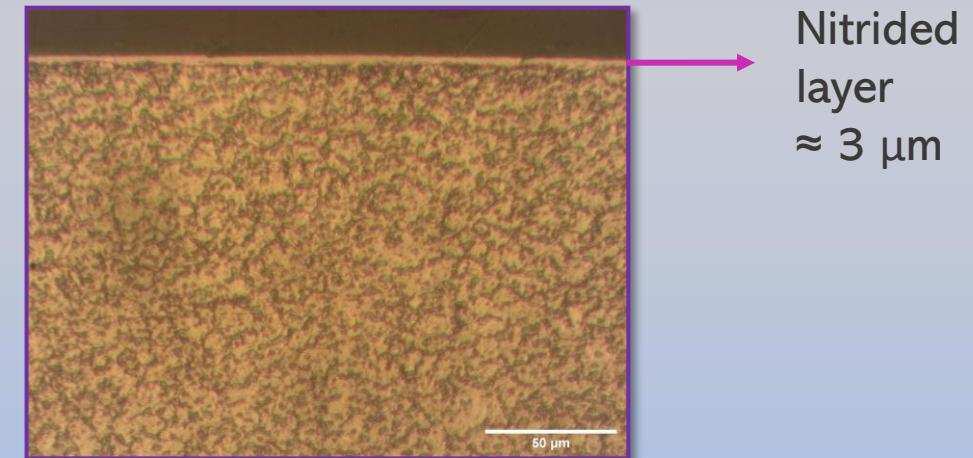
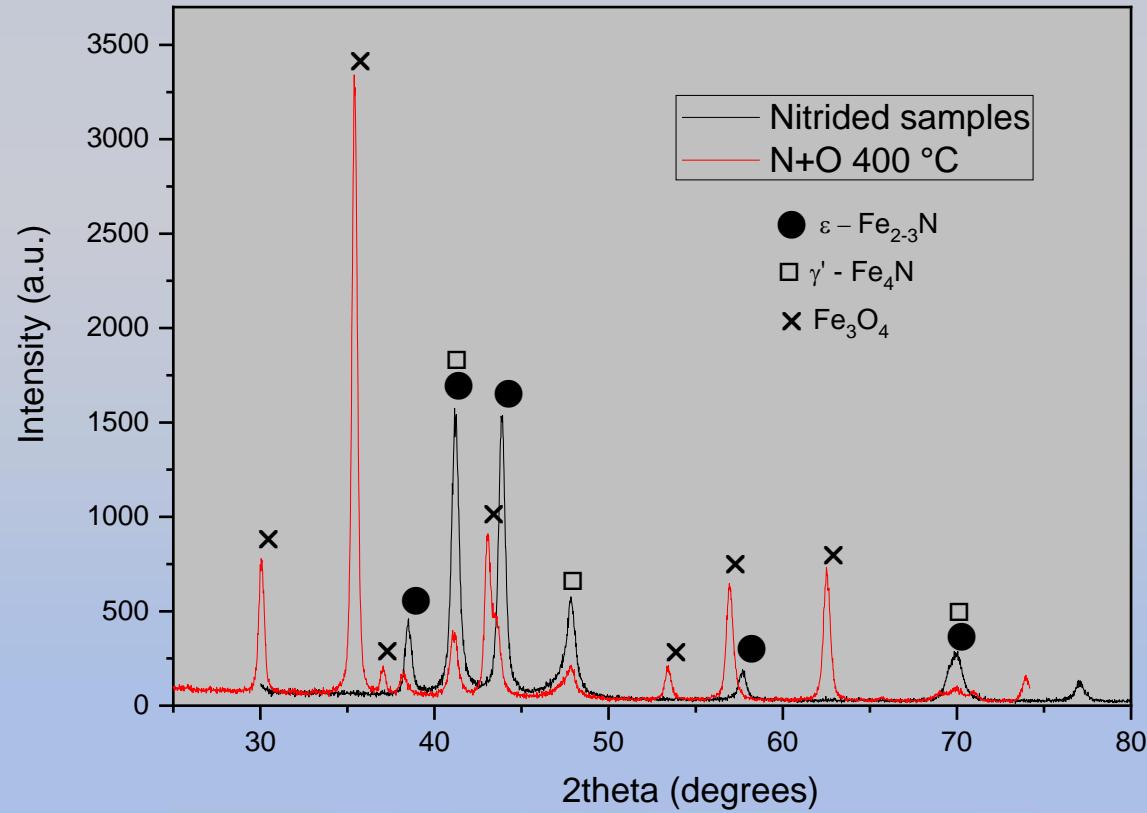


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Results

Microstructure

XRD grazing incidence 3 degrees



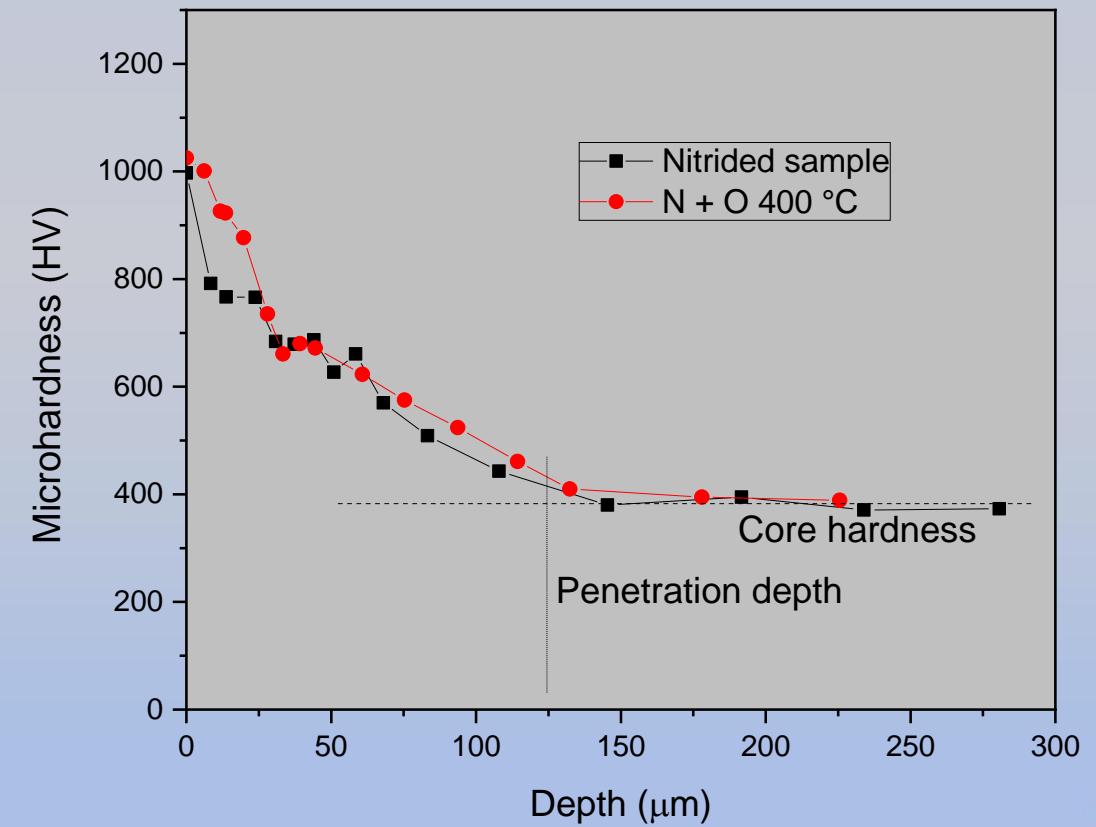
Optical micrograph
cross section N + Ox

Results

Surface Hardness and depth profile

Vickers microindenter

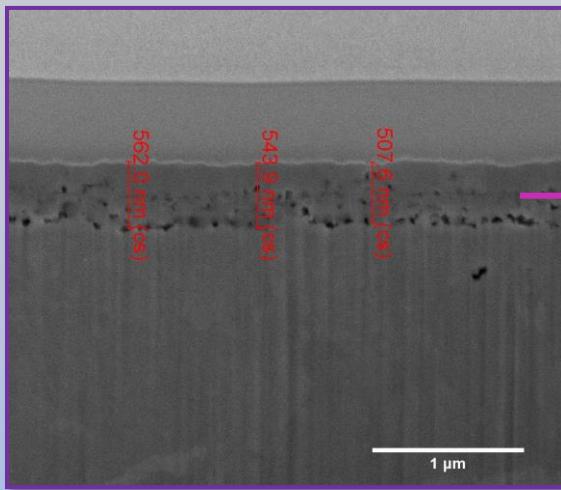
Samples	Hardness HV 0.05
Nitrided + oxidized steel	1020 ± 50
Nitrided steel	1000 ± 50
Heat treated steel	390 ± 10



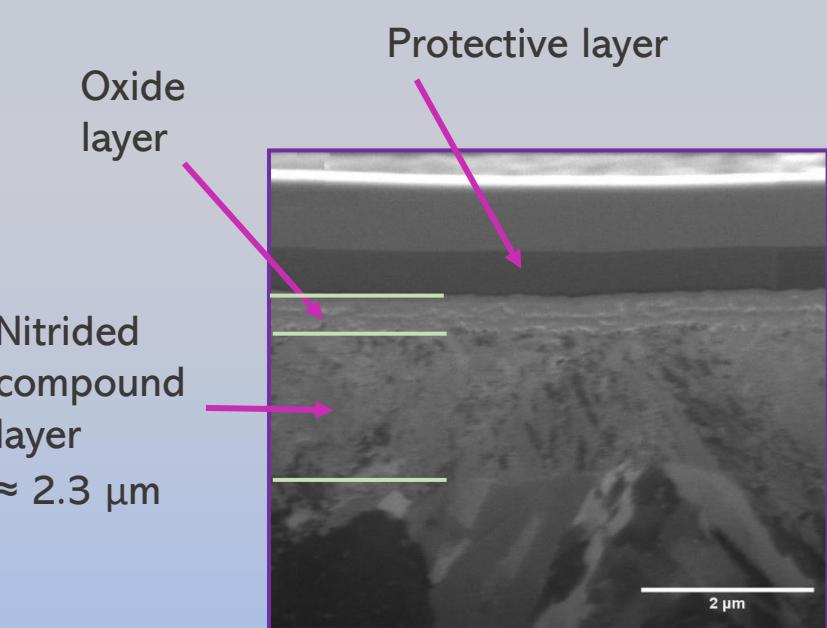
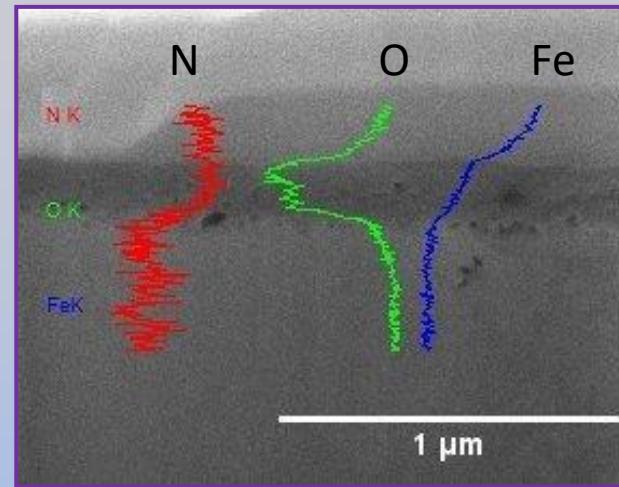
Results

Layers thickness

FIB-SEM images on different samples



Oxide
layer
 $\approx 0,5 \mu\text{m}$

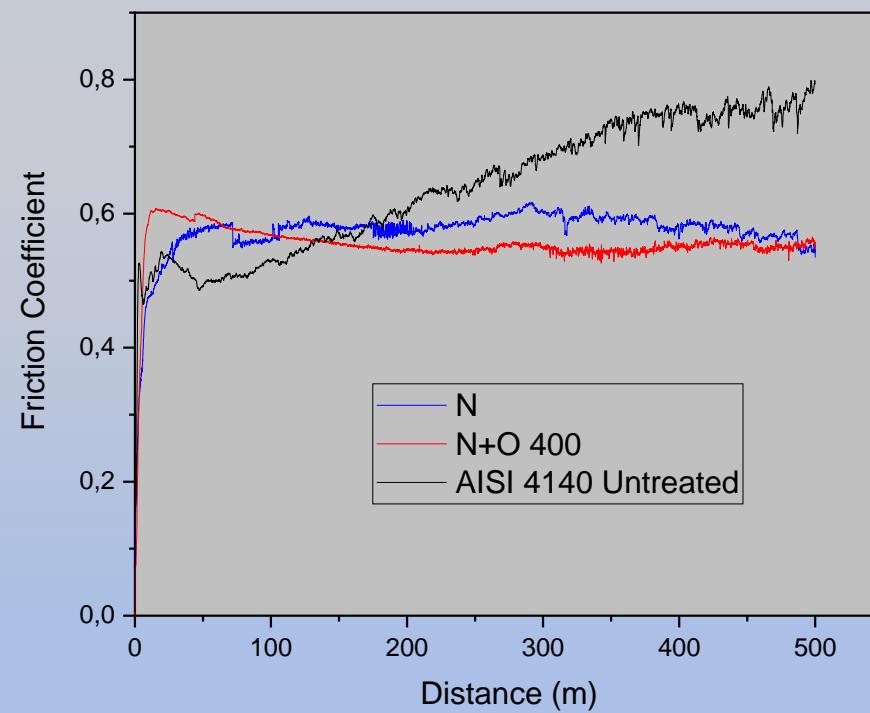


Results

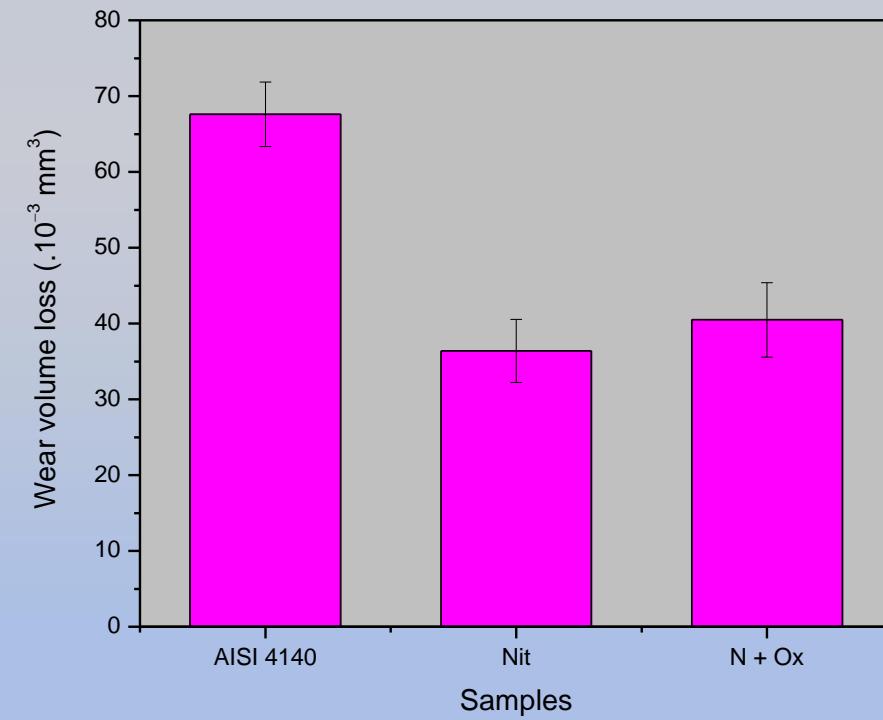
Wear and Friction

Pin on disk results

Friction coefficient



Wear volume loss



UTN
FRCU
Facultad Regional
Concepción del Uruguay

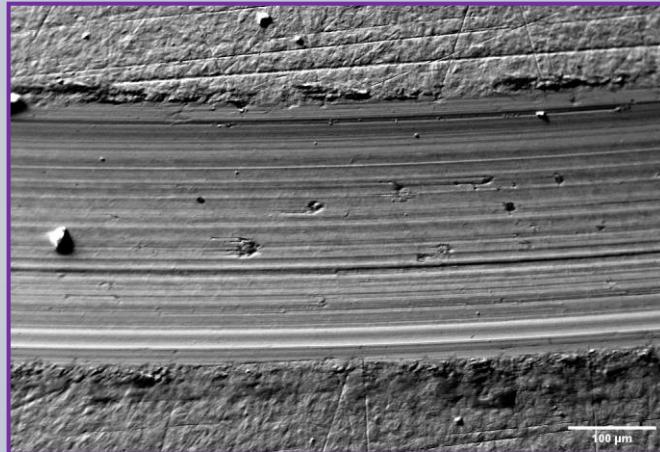
GIS
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Results

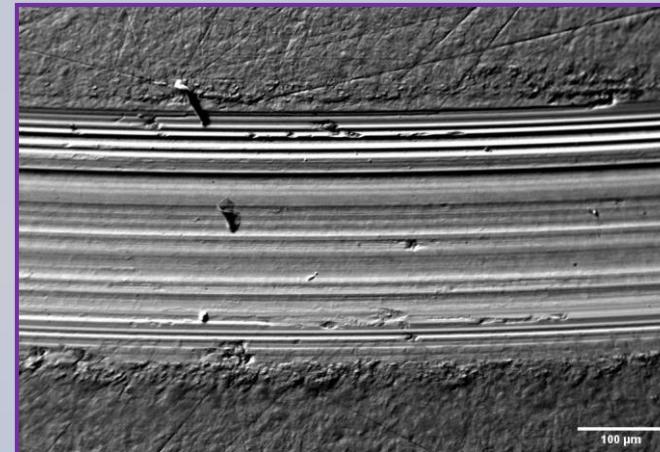
Wear tracks

Pin on disk
wear tracks

Nitrided sample



N + Ox



Counterpart



SEM
Images



OM
Images

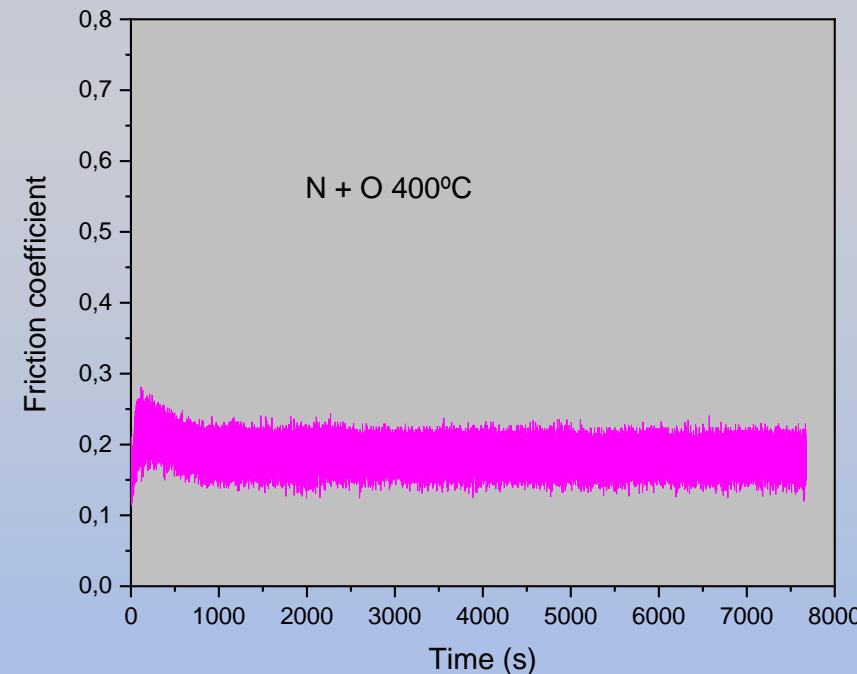
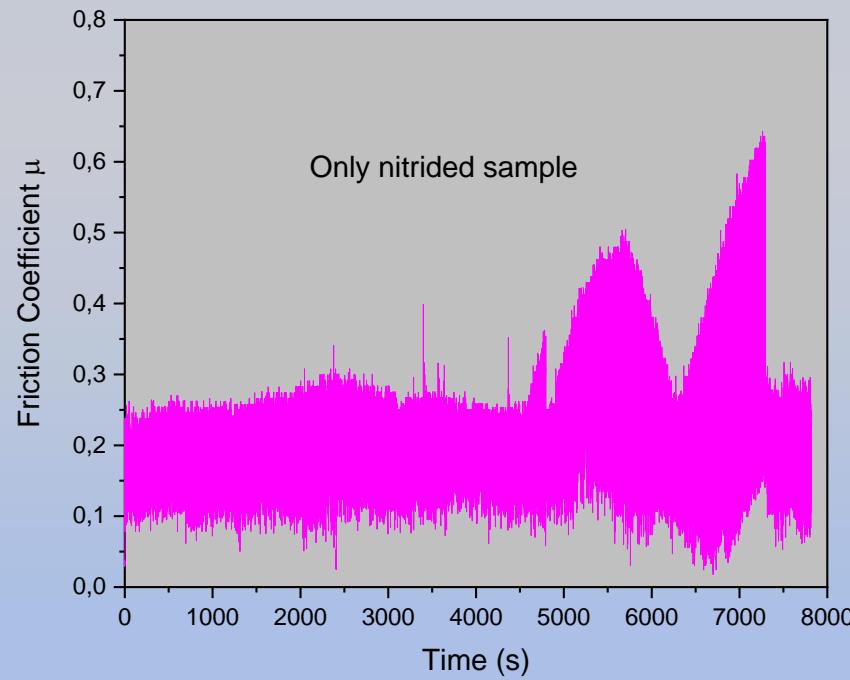


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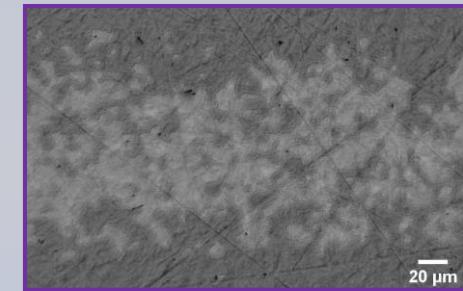
Results

Reciprocating sliding tests with lubrication

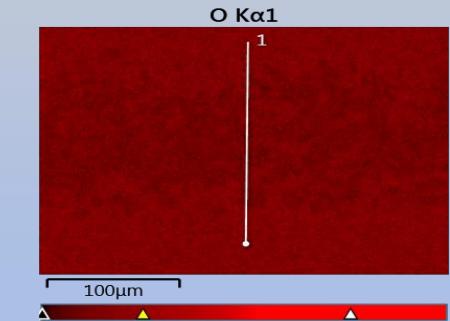
Friction coefficient



N + Ox



SEM image of the track



EDS map

Thanks to W. Tuckart and G.Prieto, UNS-CONICET, Ba. Bca. Arg

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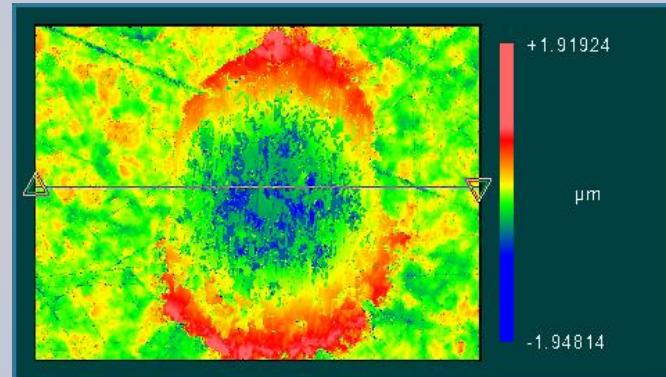
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Results

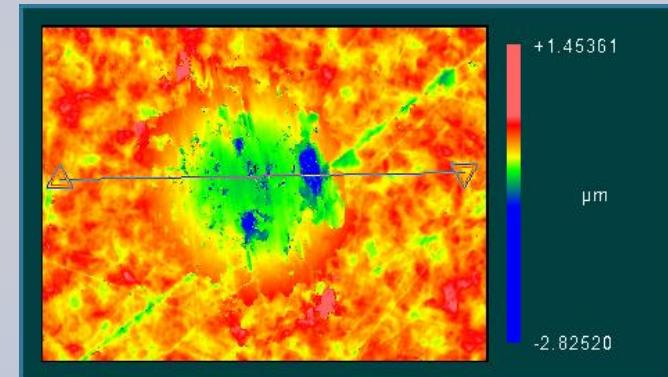
Fretting wear tracks

WLI images

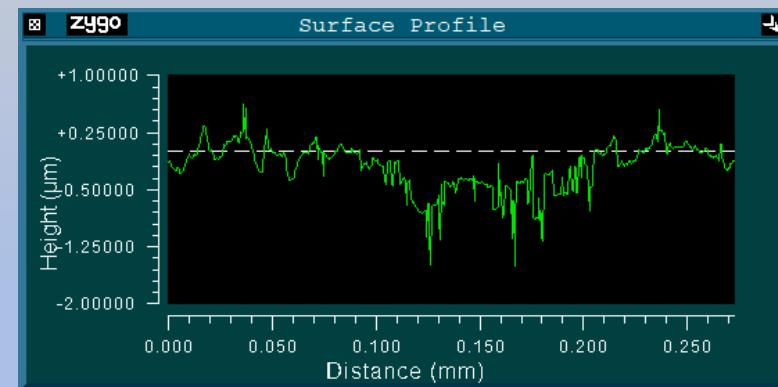
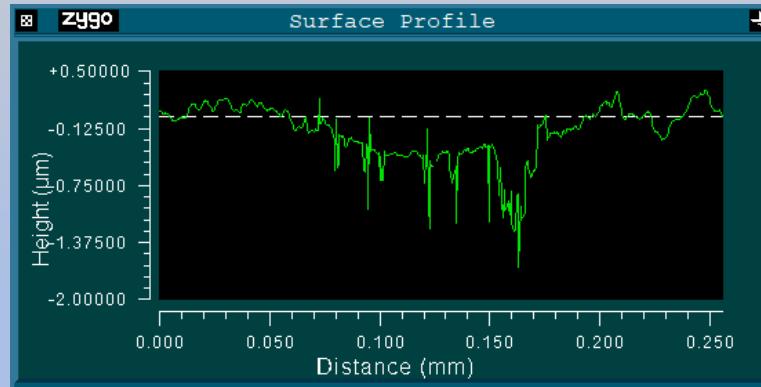
Nitrided



N + Ox



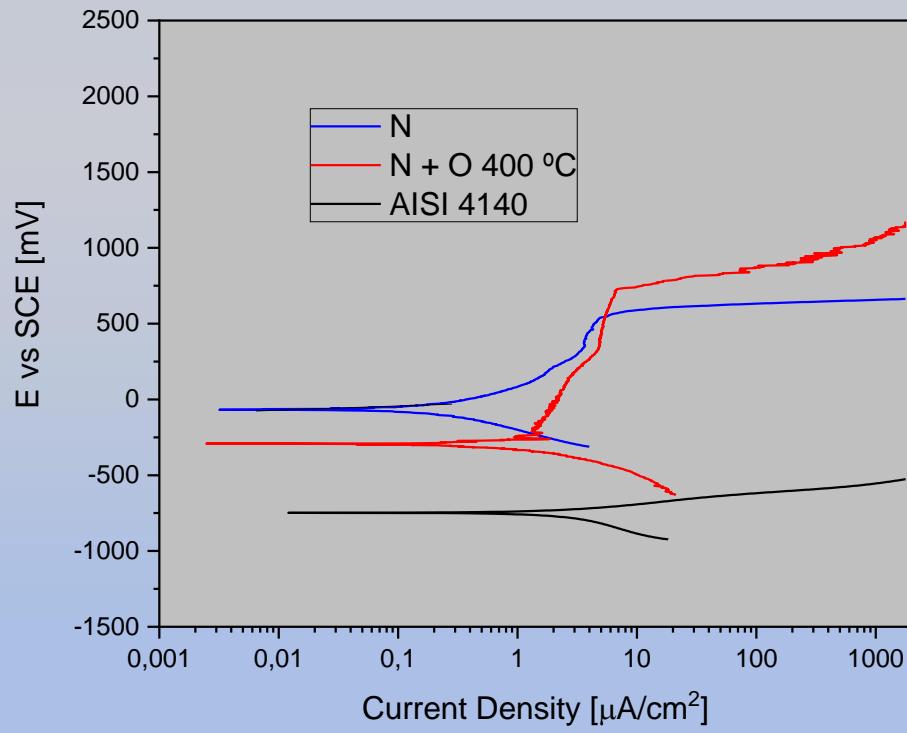
WLI Profiles



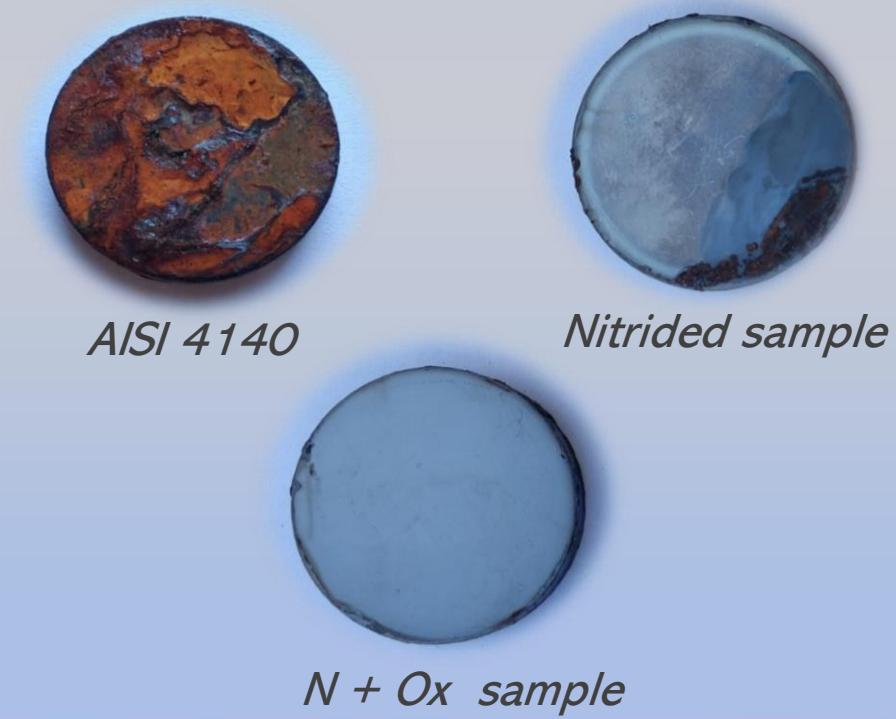
Results

Corrosion behaviour

Potentiodynamic polarization in NaCl 3.5 %



Salt Spray Fog Test results



Results

Corrosion morphology

Optical micrographs

Crevice + pitting

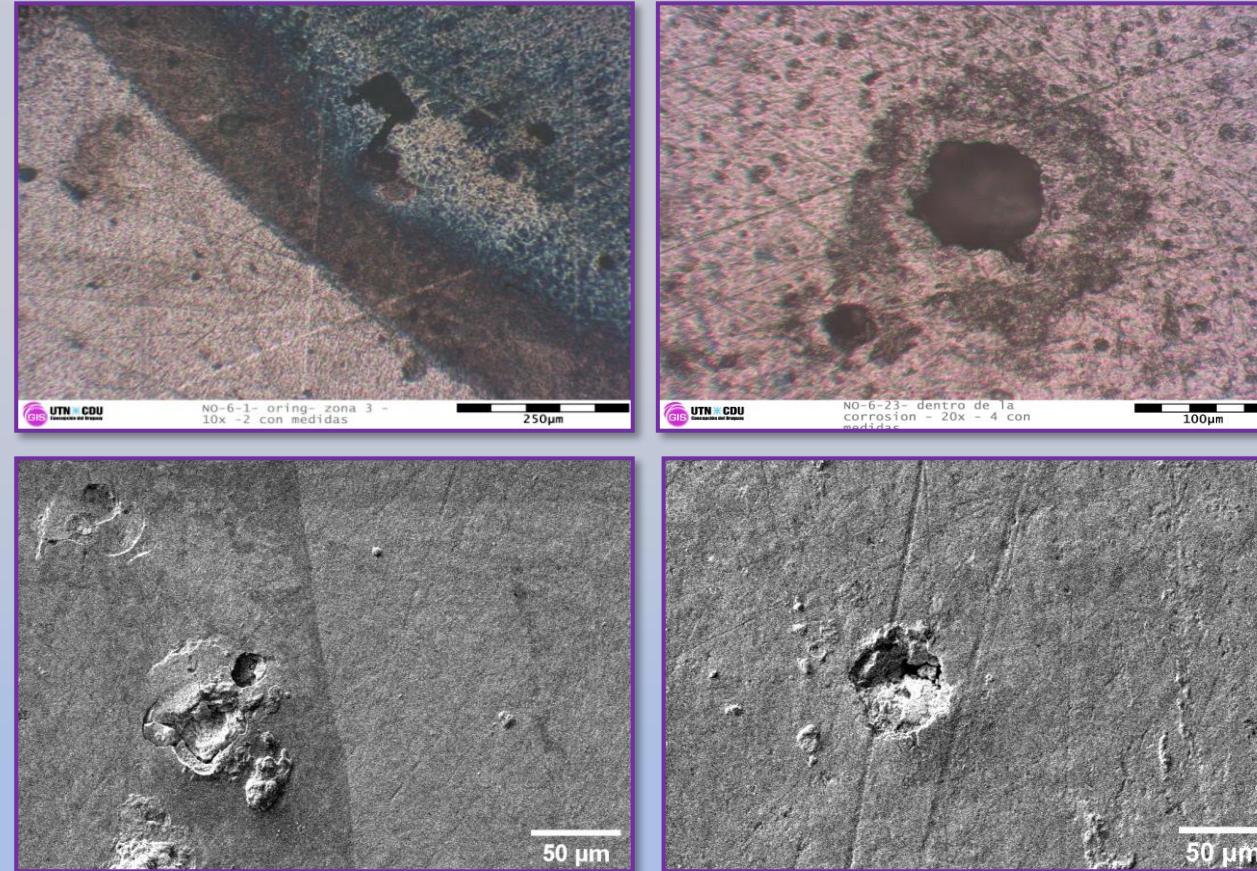
SEM micrographs

Nitrided

N + Ox



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Pitting

Conclusions

1. A duplex plasma treatment, nitriding + oxidizing, was applied successfully in the same process work chamber over AISI 4140 steel.
2. An oxide layer of 0.5 µm thick was formed after the oxidizing treatment, consisting in pure magnetite.
3. This layer did not improve the tribological behaviour under dry conditions (pin on disk and fretting) in comparison with the nitrided layer. However, it reduced the friction coefficient in reciprocating sliding tests under lubricated conditions and low hertzian pressure.
4. The oxidized layer, provided good corrosion protection in saline environments as revealed in the salt spray and potentiodynamic tests.



Thank you!



sonia@frcu.utn.edu.ar



Sonia Brühl



<http://www.frcu.utn.edu.ar/gis>



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¡Muchas Gracias!