Carbon based coatings deposited over AISI 4140 to improve wear resistance in machine components

Francisco Delfín*, Sonia Brühl

Surface Engineering Group
National University of Technology (UTN)
Faculty of Concepción del Uruguay
Argentina

Daniel Heim

* Upper Austria University of Applied Sciences
Wels Campus
Austria

ICMCTF 2023

San Diego, USA

22-26 May 2023

UTN FRCU
GIS 25 AÑOS
Grupo de Ingeniería de Superficies
1998-2023

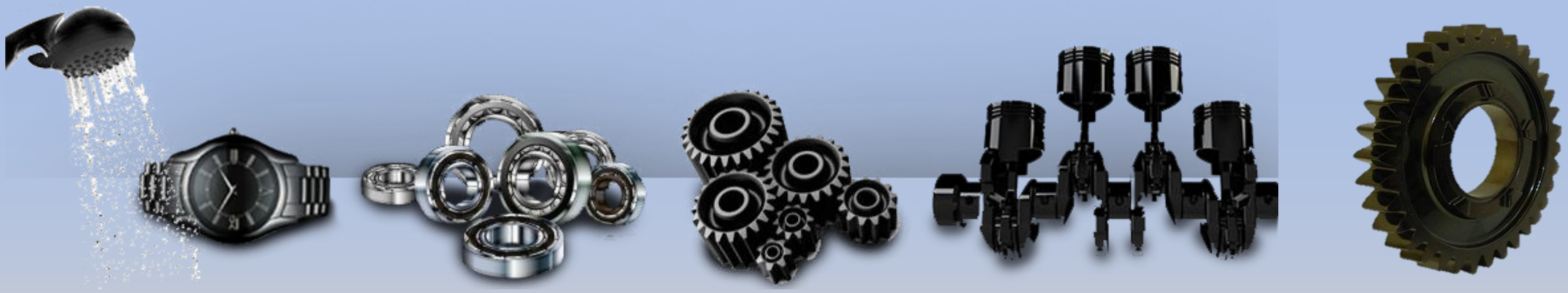
FH Oberösterreich
UNIVERSITY OF APPLIED SCIENCES UPPER AUSTRIA

Carbon Based Coatings

DLC Diamond-like Carbon

High hardness, low friction coefficient, chemical inertia

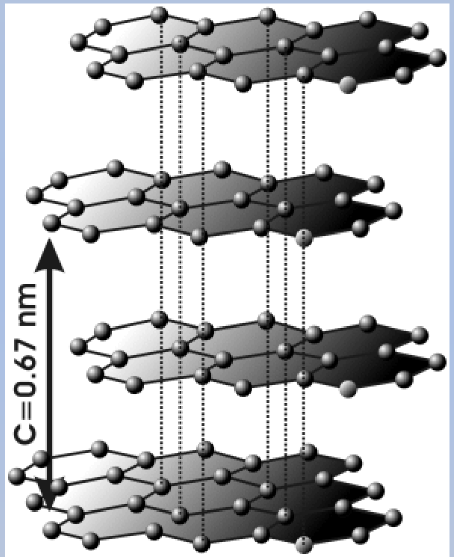
Good resistance to wear and corrosion



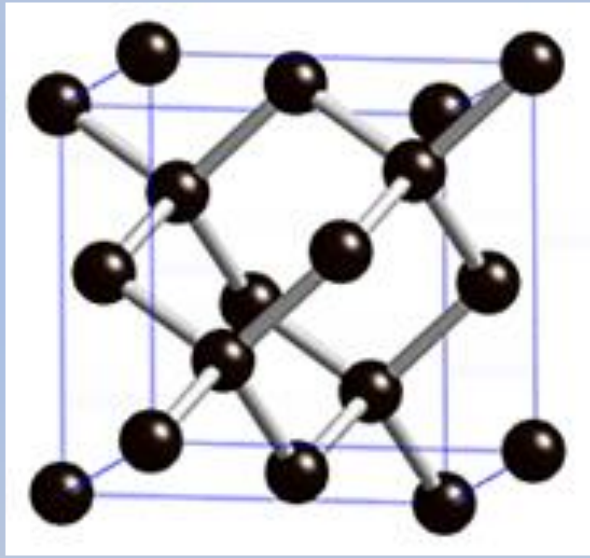
Carbon Based Coatings

DLC Diamond-like Carbon usually produced by thermal CVD, PACVD and PVD

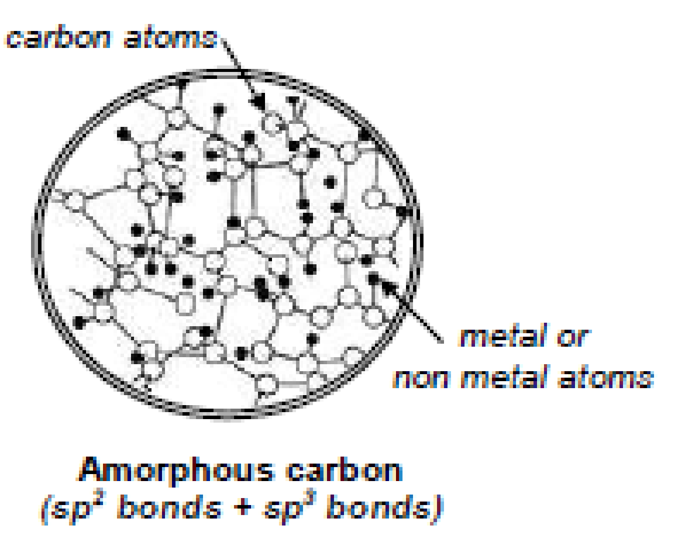
Graphite, pure sp2 bondings



Diamond, pure sp3



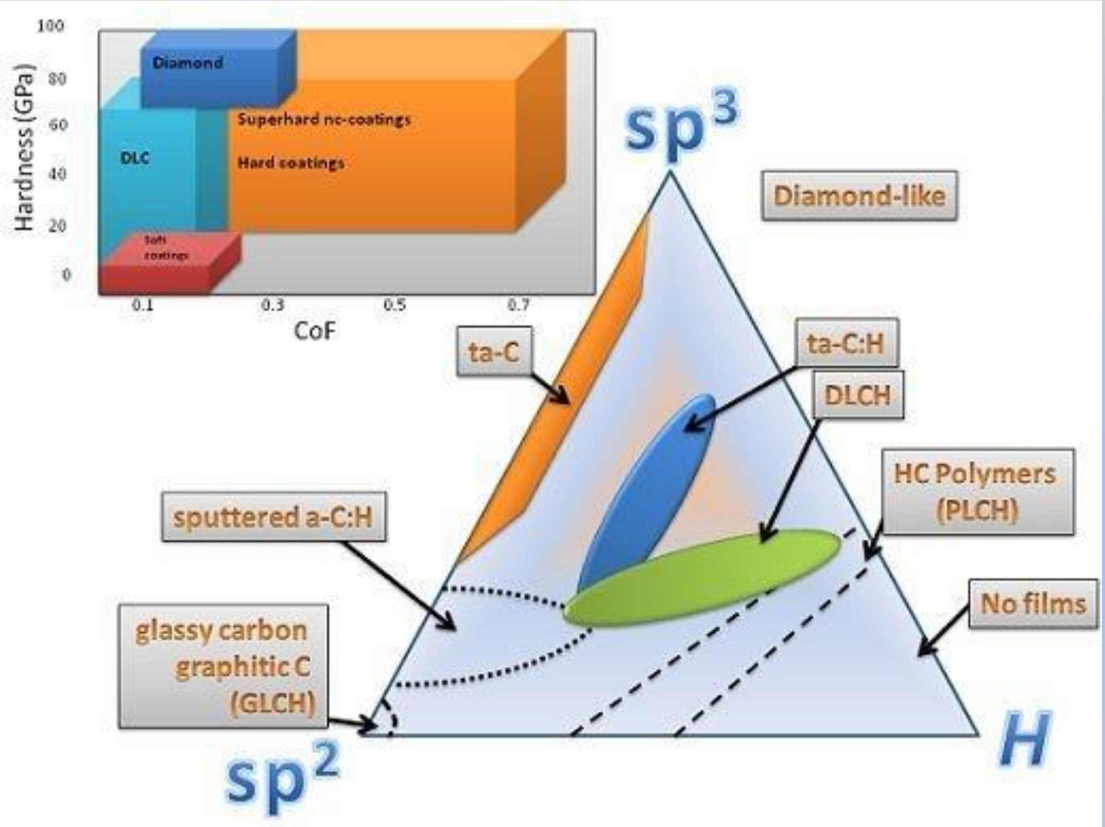
DLC



Different DLC coatings

Examples of different DLC coatings properties

Film Type	% H	sp3 bondings (%)	Hardness (HV)	Typical width (µm)
ta-C	< 1	85-95	4000-13000	2- 4
Hard a-C:H	10-40	30-60	1000-4000	2- 4
Soft a-C:H	40-65	50-80	<1000	20- 50



Lubricants **2013**, *1*(2), 22 47

Main Goals and Outline

- To present two different DLC films deposited onto SAE 4140 steel
- To analyse the wear behaviour of a DLC as top coating over a Cr/CrN multilayer
- To analyse the influence of Si doping on tribological properties

1. Experimental – Samples and characterization/testing Tribological behaviour
2. Adhesion and Abrasion resistance
3. Discussion and Conclusions

Experimental

Samples and Pre treatment

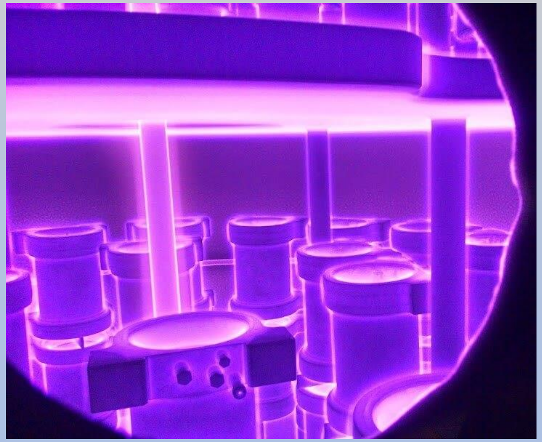
DLC coatings Nr. 1

Samples: AISI 4140 steel (DIN 1.7225)

- Discs 25 mm cut form a HT rounded bar
- Grounded with SiC until #1000 in one face

Ion nitriding pre treatment

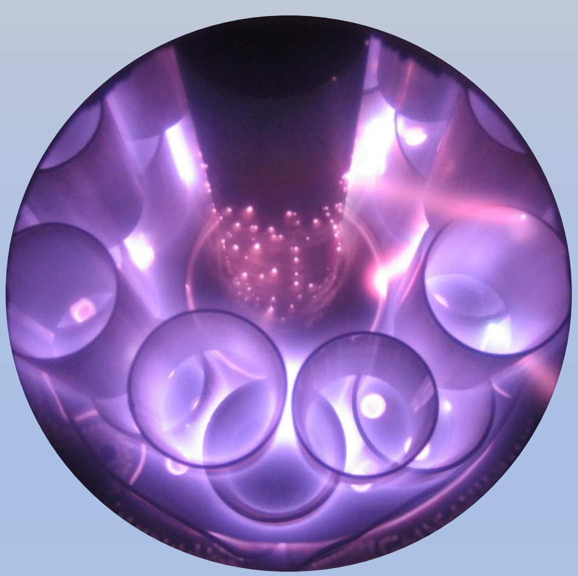
- N_2 25% - H_2 at 500 °C, 20 h
- IONAR S.A. Argentina



Experimental

DLC coatings Nr. 1 – PVD PEMS

- Cr and Graphite target with N and C_2H_2 as reactive gases
- Metallic Cr adhesion layer
- CrN as anchor layer
- Cr compounds gradient CrN/CrCN/CrC
- Deposition of top layer Cr dopped DLC (a-C:H:Cr)



Cemecon CC800 DC Sputter at COATING.TECH BY TANTAL-FLUBETECH Argentina

Experimental

DLC coatings Nr. 2 – PACVD

- 2 mbar pressure
- C_2H_2 + Ar
- 150 W power
- Process duration: 35 h
- Temperature: 450 °C
- Si incorporation using HDMSO

Silicon content

2.1 Si Free (a-C:H)

2.2 Si Cont. (a-C:H:Si)

Tribotest Atmosphere air

- Dry 4% RH
- Room 20% RH
- Wet 80% RH

GIS Grupo de Ingeniería de Superficies, UTN FRCU, 25 años, 1998-2023

Modified
Rübig GmbH facility in

Fh OBERÖSTERREICH UNIVERSITY OF APPLIED SCIENCES UPPER AUSTRIA

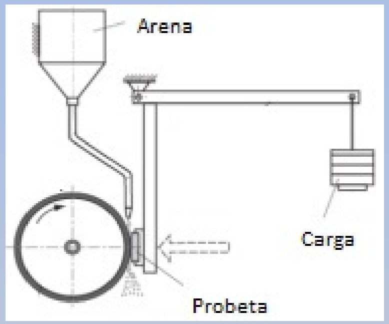
Characterization and tests

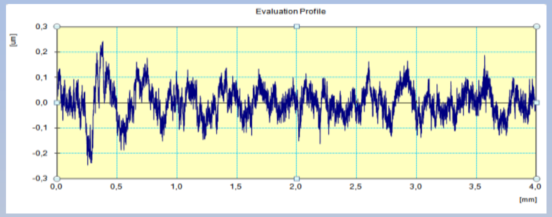
UTN FRCU 1998-2023 GIS 25 AÑOS Grupo de Ingeniería de Superficies

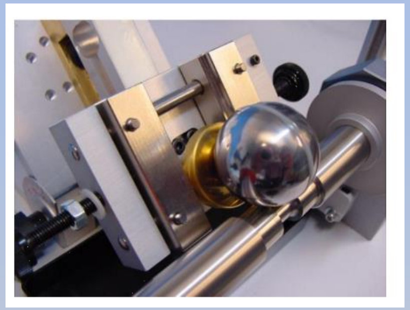
1) Microstructure: SEM, EDS, Raman
   Roughness, Calotest, Nanoindentation

2) Wear tests: adhesive ASTM G-99 Pin on Disk - rotational
   abrasion ASTM G-65 Dry Sand Rubber wheel

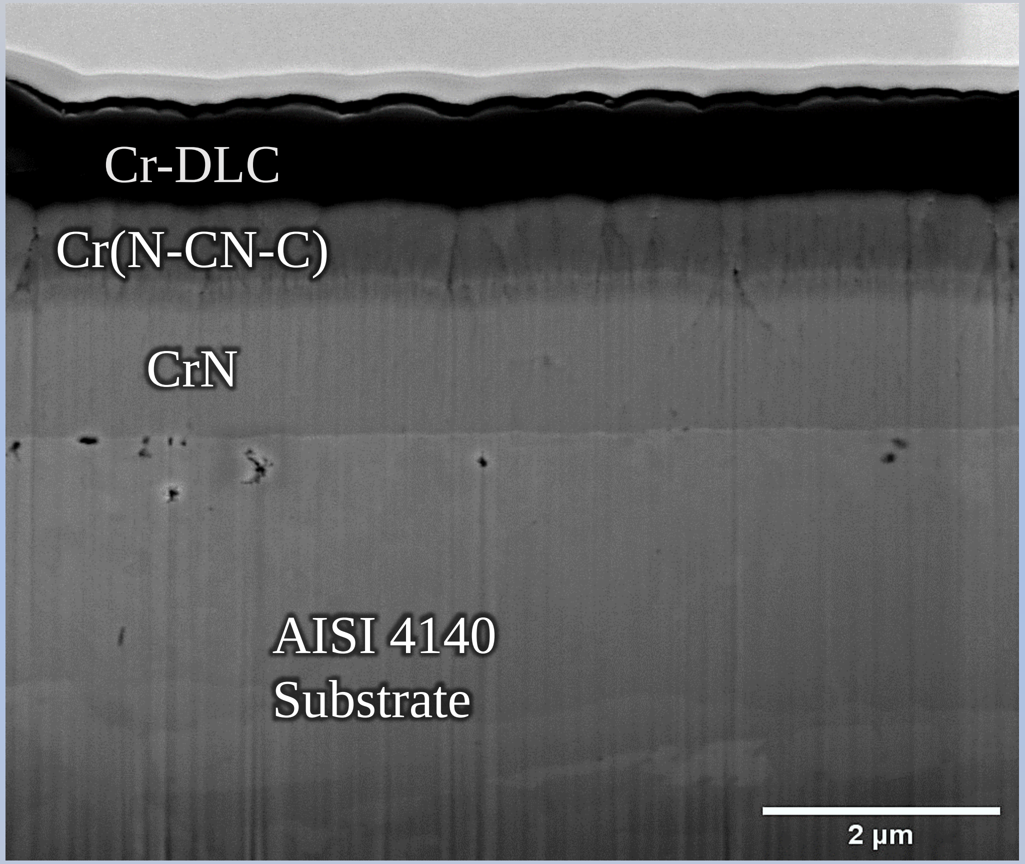
3) Adhesion: Scratch Test, Rockwell C Indentation







DLC 1- Results - Characterization



Mechanical Properties:

Young Modulus: 143 ± 9 GPa

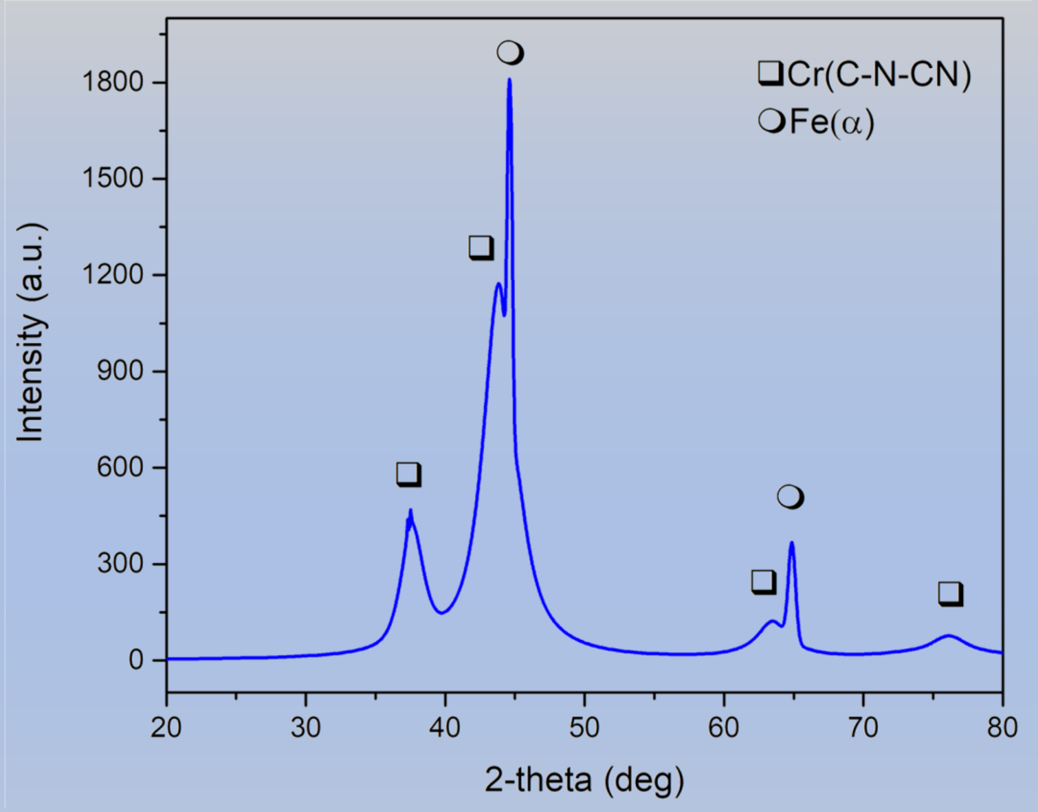
Hardness: 12 ± 1 GPa (1150 ± 120 HV)

Thickness:

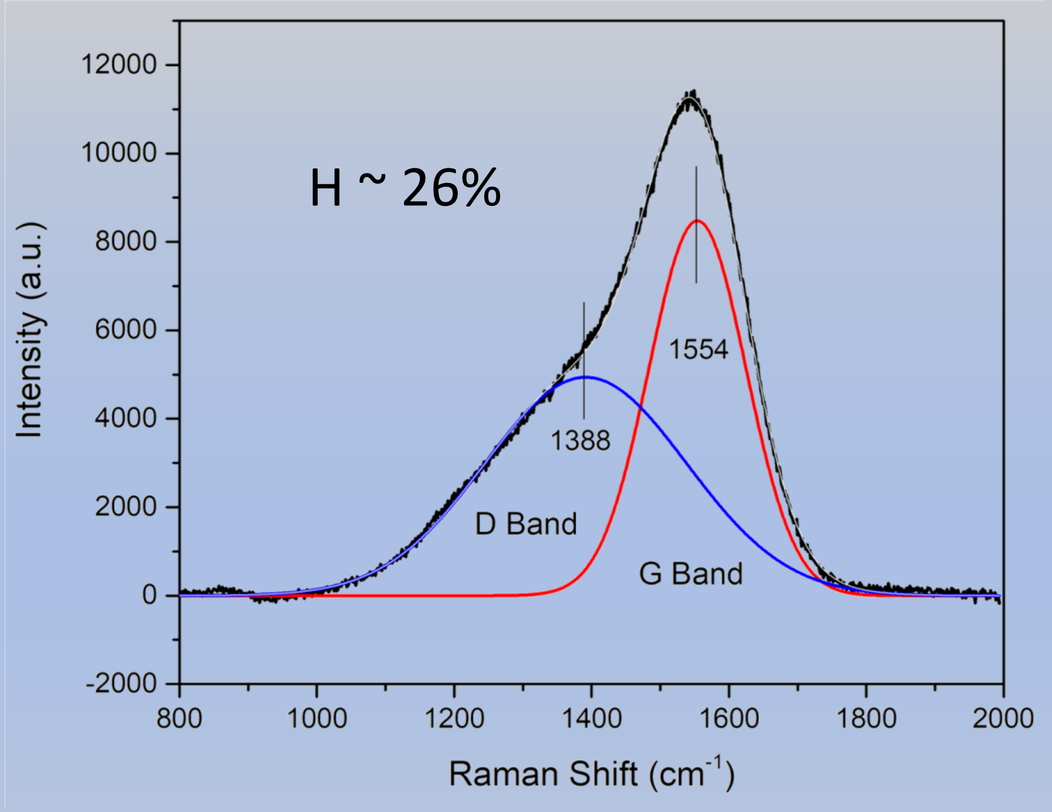
Total: 4.1 ± 0.2 µm

Only Cr-DLC Layer: 1.4 ± 0.1 µm

DLC 1- Results - Characterization



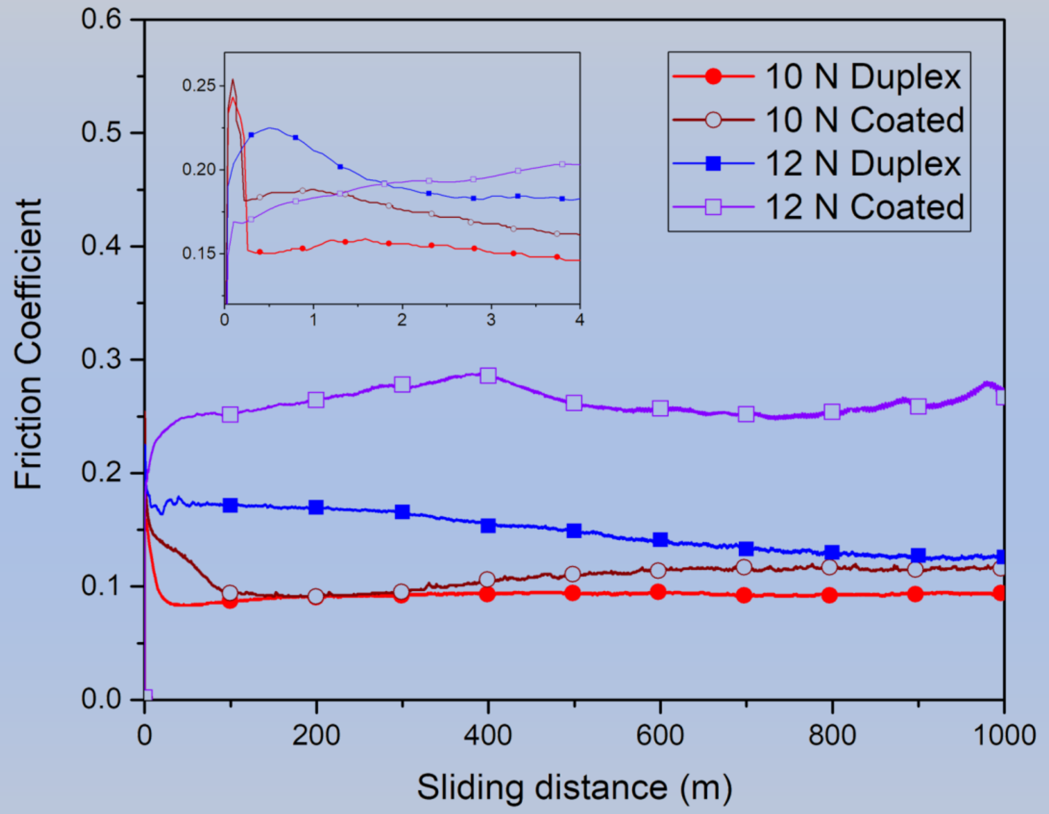
X-Ray Diffraction Pattern

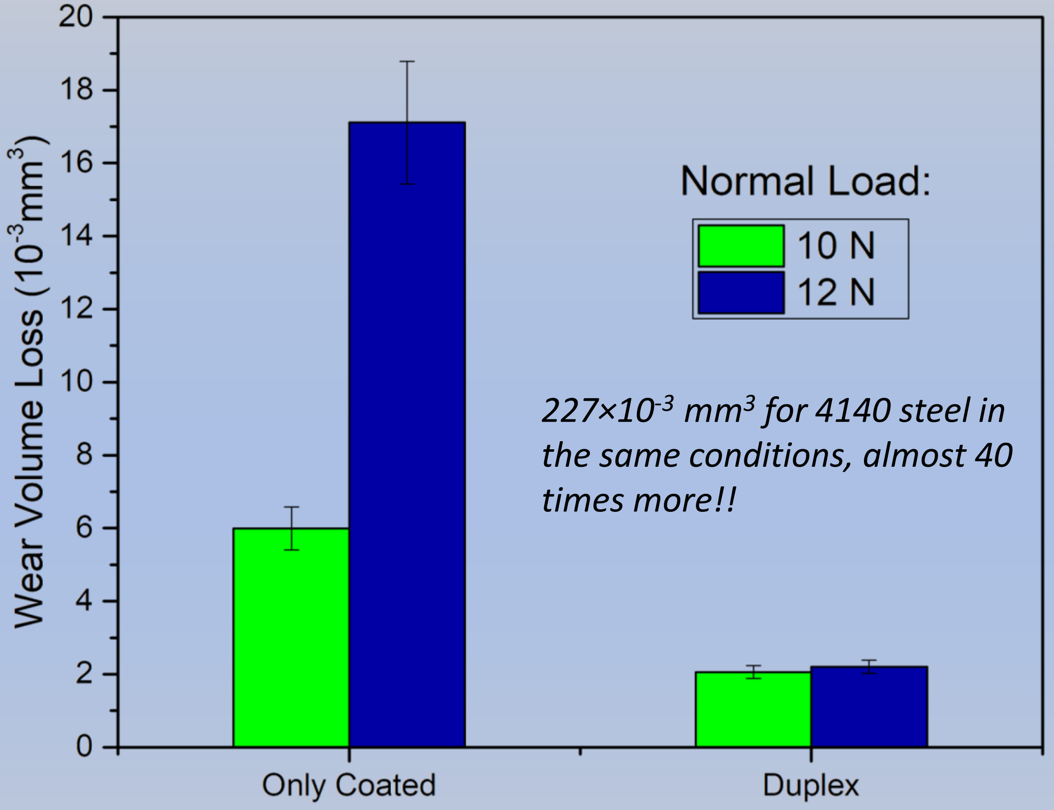


Raman Spectra

DLC 1- Results – Tribology, wear

Counterpart Al_2O_3 Ø 6 mm; Sliding Distance 1000 m; Normal Load 10 N





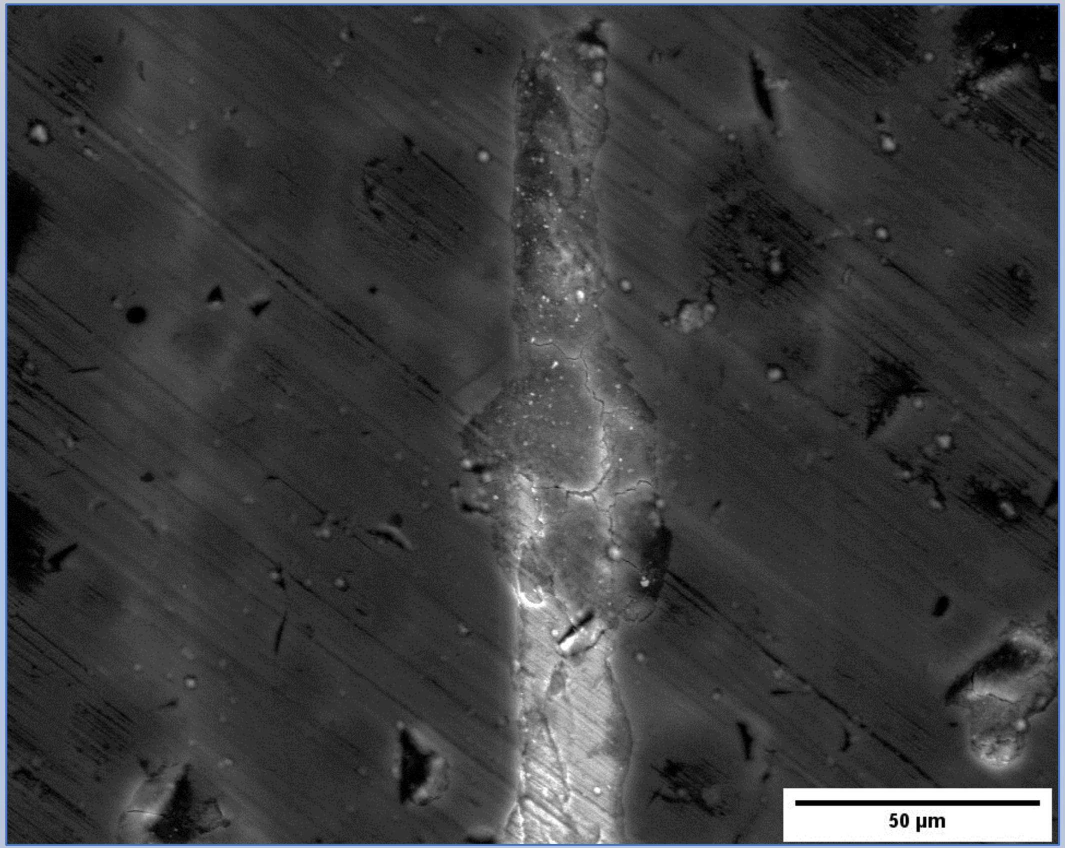
227×10⁻³ mm³ for 4140 steel in the same conditions, almost 40 times more!!

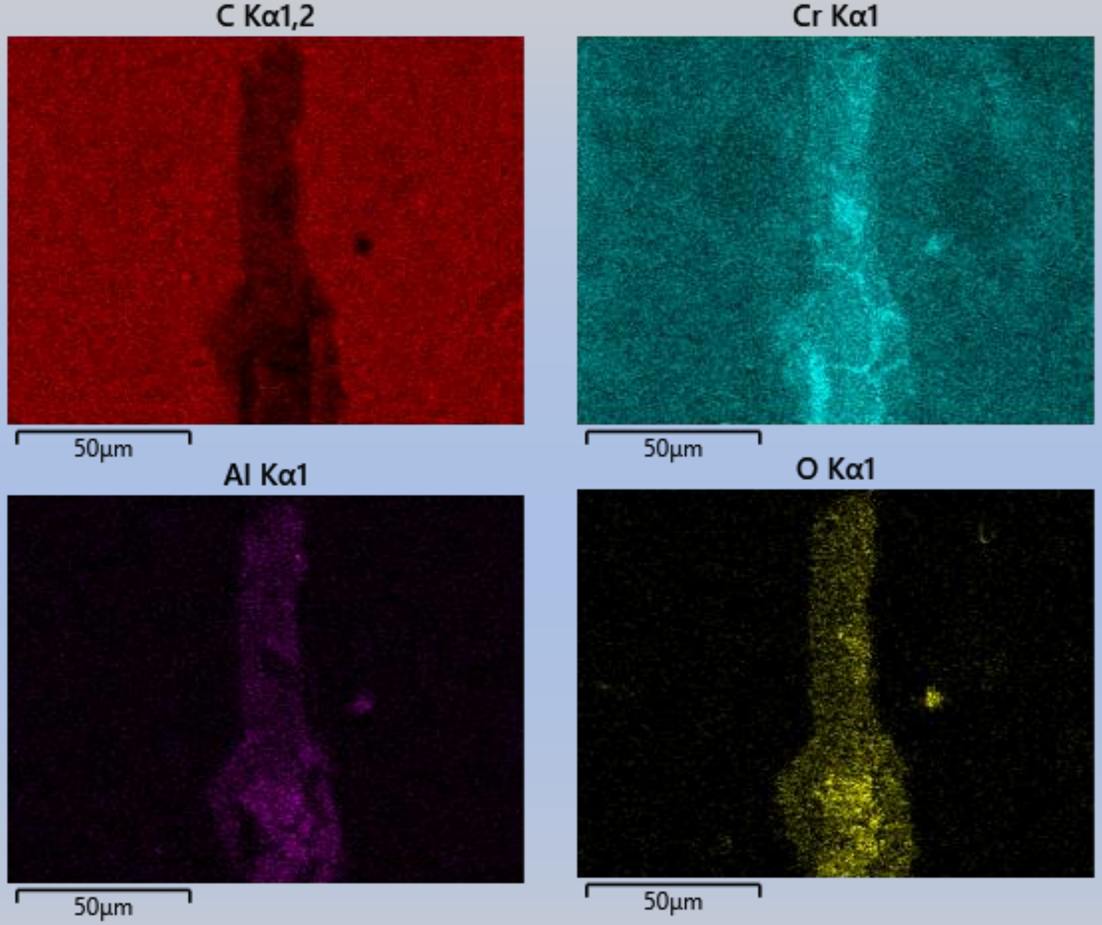
Average environment conditions: 20 °C, 60% RH

DLC 1- Results – Tribology, wear

UTN FRCU 1998-2023 GIS Grupo de Ingeniería de Superficies 25 AÑOS

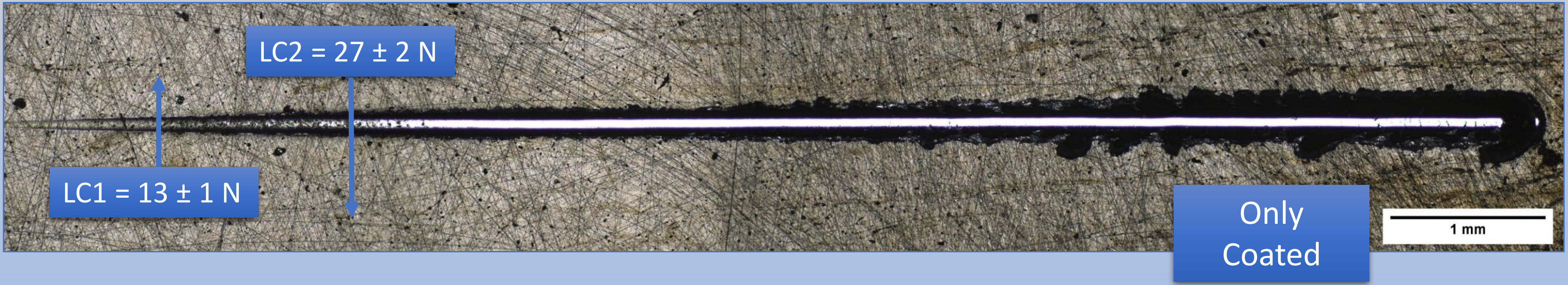
SEM Analysis with EDS mapping of the worn track

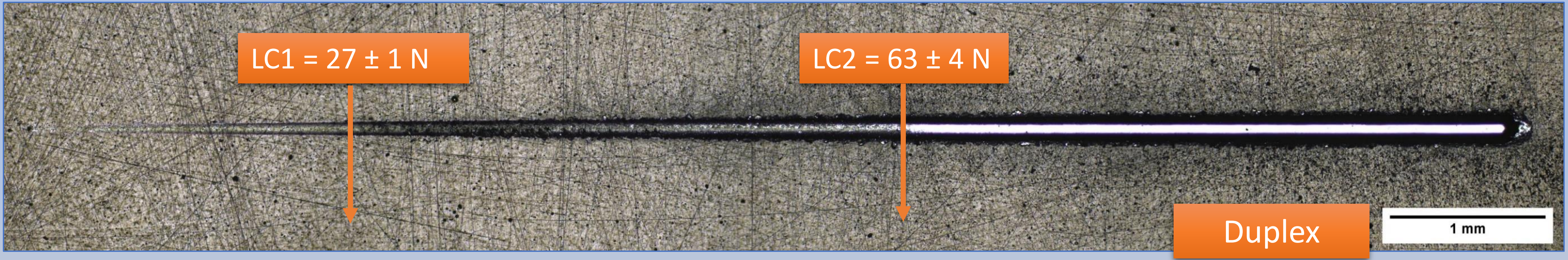




DLC 1- Results - Adhesion

Variable Load Scratch Test in the range from 1 to 101 N





DLC 1- Partial Conclusions

- ✓ Cr/CrN/DLC Multilayer coating with excellent wear resistance.
- ✓ Low friction coefficient provided by the DLC as top coating.
- ✓ The only coated samples presented higher wear due to poor adhesion.
- ✓ Nitriding as pre treatment improves the load bearing capacity and adhesion of the coating.

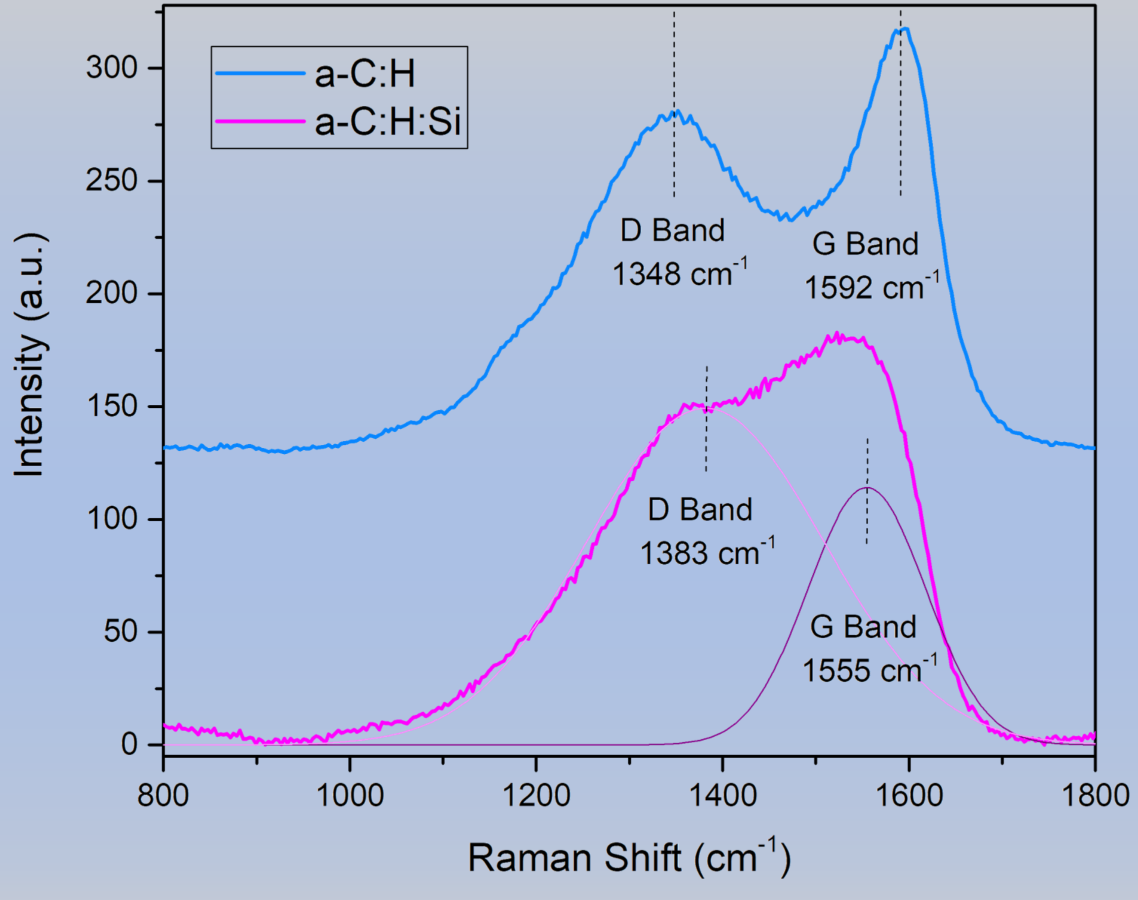
DLC 2 - Results - Characterization

Si free (a-C:H) coating:

- D y G bands close to reference values.
- I_D/I_G relation = 0.87

Si cont. coating (a-C:H:Si):

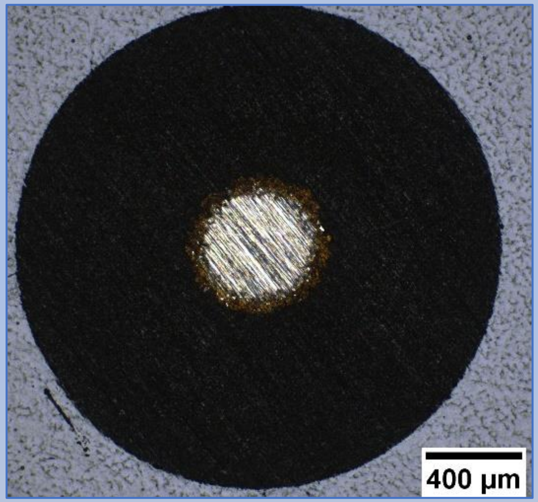
- D y G bands closer to the center. Higher level of disorder.
- I_D/I_G relation = 1.31

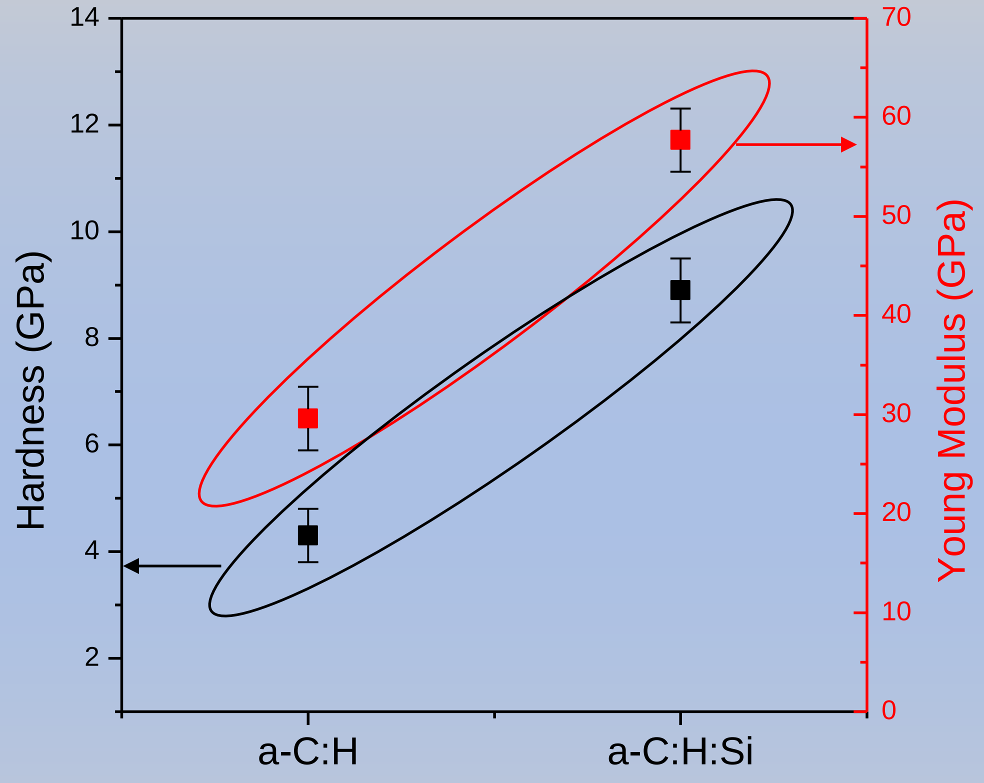


DLC 2 - Results - Characterization

Coating thickness (calotest)

- a-C:H = 54 ± 4 μm
- a-C:H:Si = 42 ± 1 μm





Hardness and Y Modulus doubled when inserting Si

Young Modulus:

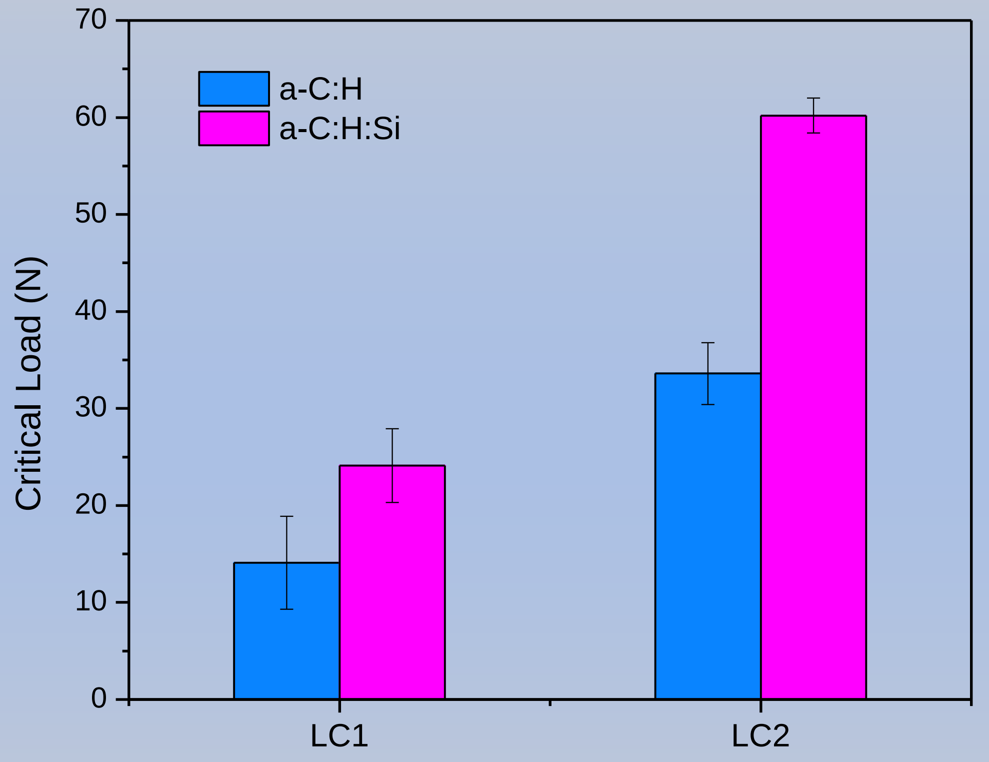
- a-C:H 30 ± 3 GPa
- a-C:H:Si 58 ± 3 Gpa

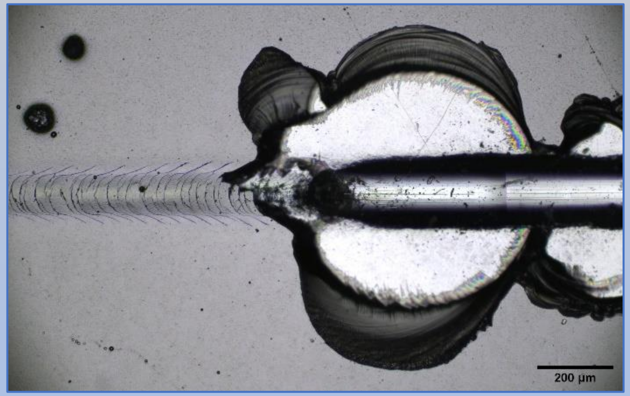
Surface Hardness:

- a-C:H 4.5 ± 0.5 GPa
- a-C:H:Si 8.9 ± 0.6 GPa

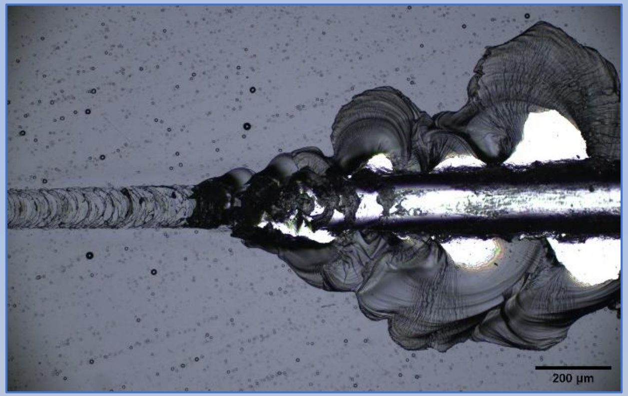
DLC 2 - Results - Adhesion

Scratch Test: Influence of the Si content





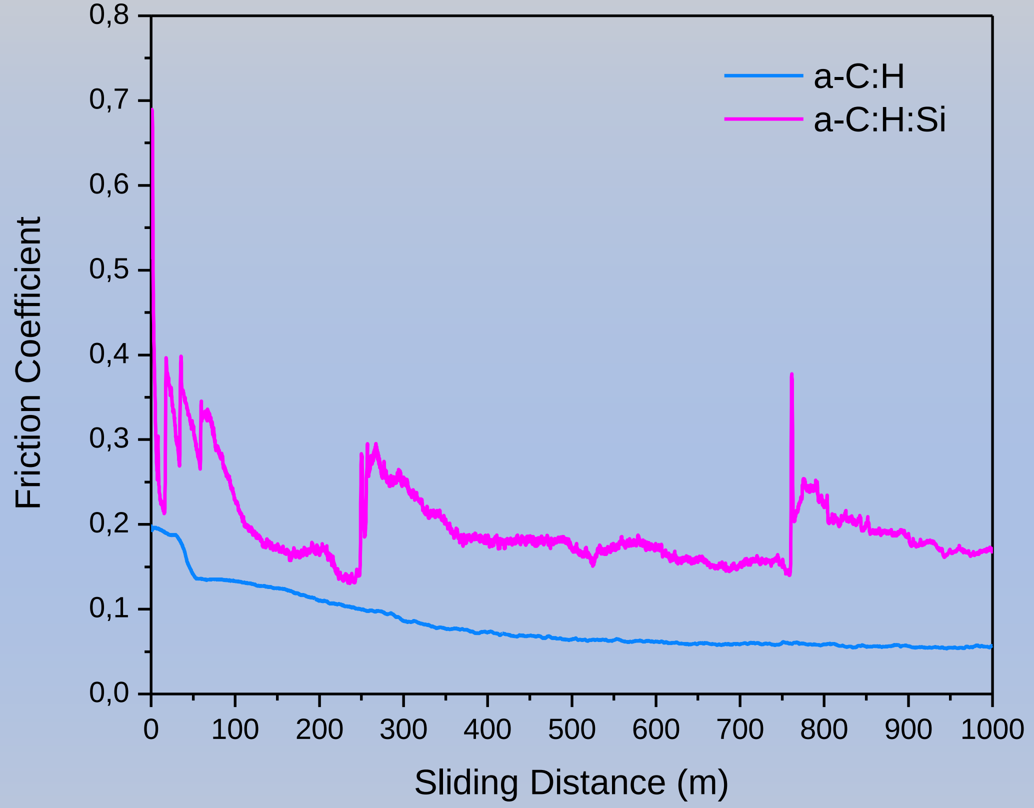
a-C:H

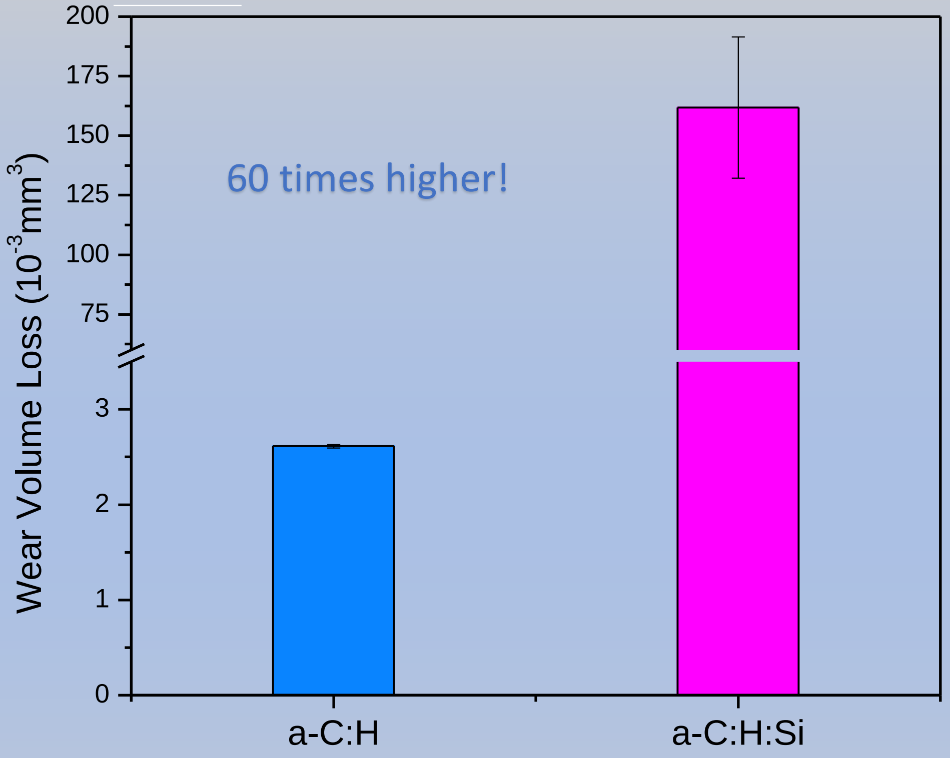


a-C:H:Si

Critical Load (L_{C2})

DLC 2 - Results - Tribology



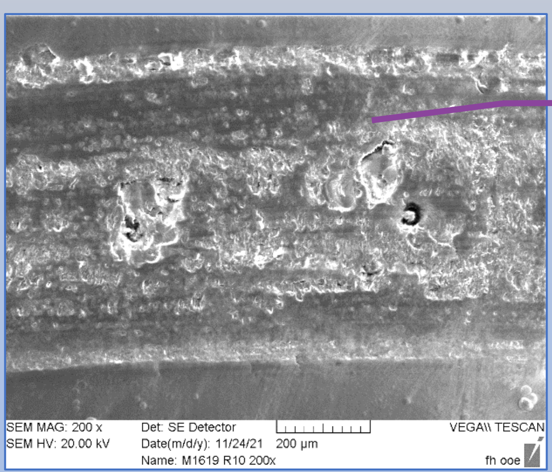


Environmental conditions: Temperature = 25 °C and Humidity = 20 %

DLC 2 - Results - Tribology

Pin-on-Disk Test wear scars: Influence of Si

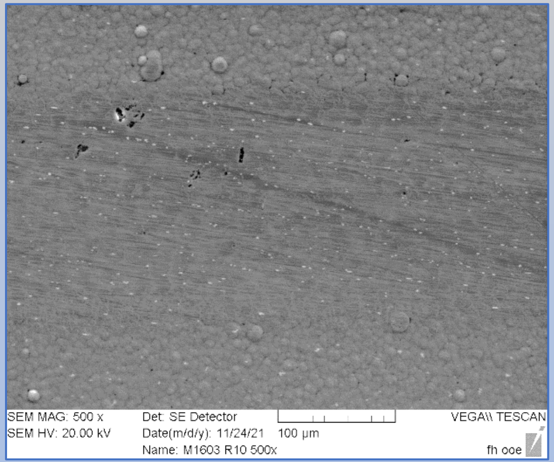
a-C:H:Si

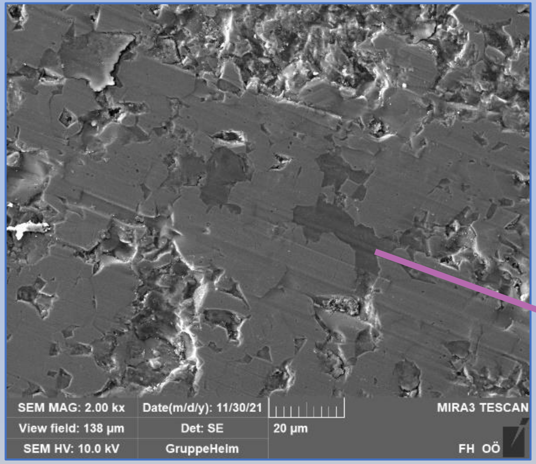


C = 46.8 %
O = 40.7 %
Si = 10.6 %
Al = 2.0 %

EDS debris analysis:
C = 46.8 %
O = 34.6 %
Si = 16.3 %
Al = 2.3 %

Si Free





Alumina
Counterpart
EDS

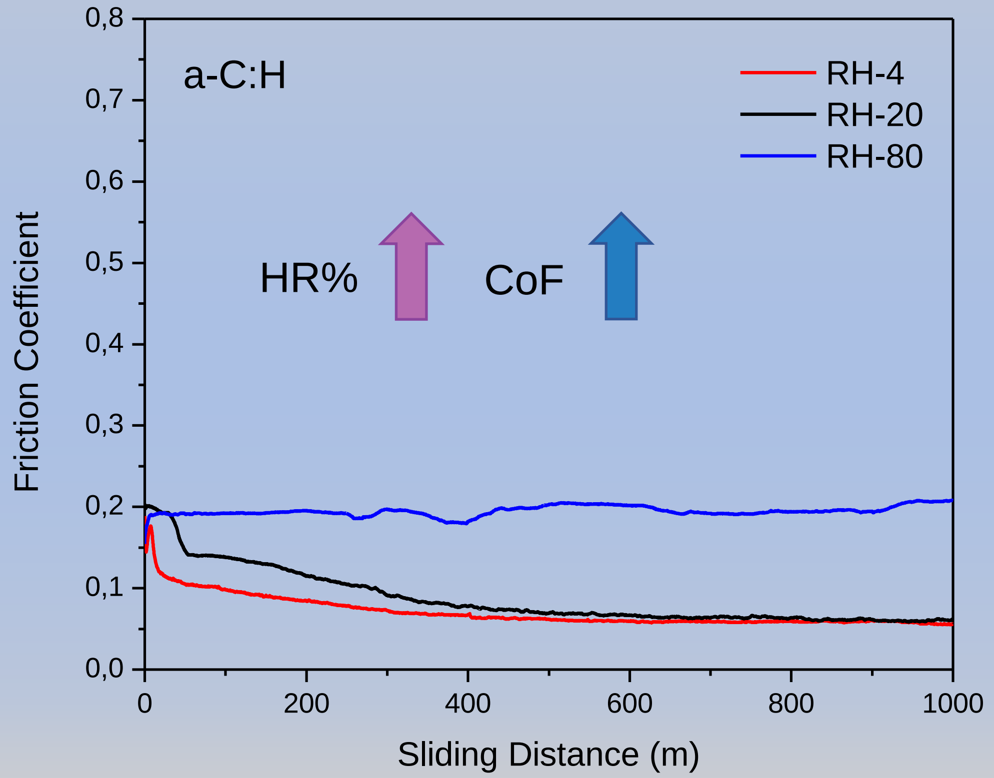
C = 54.5 %
O = 34.4 %
Si = 10.2 %
Al = 1.0 %

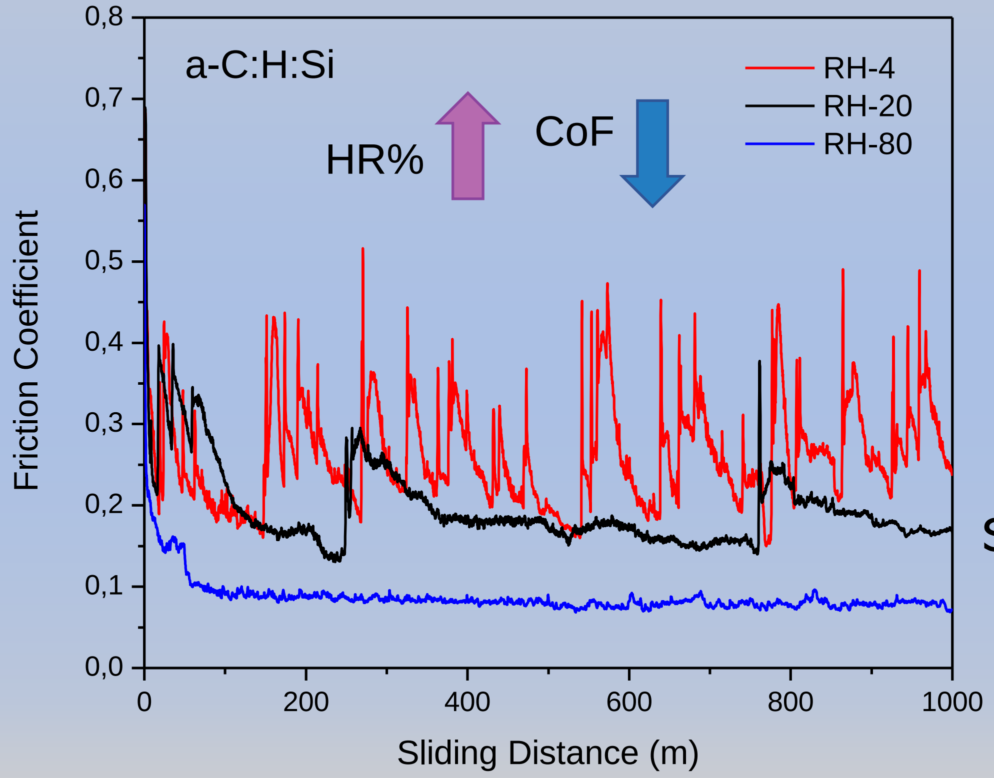
DLC 2 - Results - Tribology

Pin-on-Disk tests

Influence of relative humidity on the CoF T_{amb} ~ 25 °C

Si free



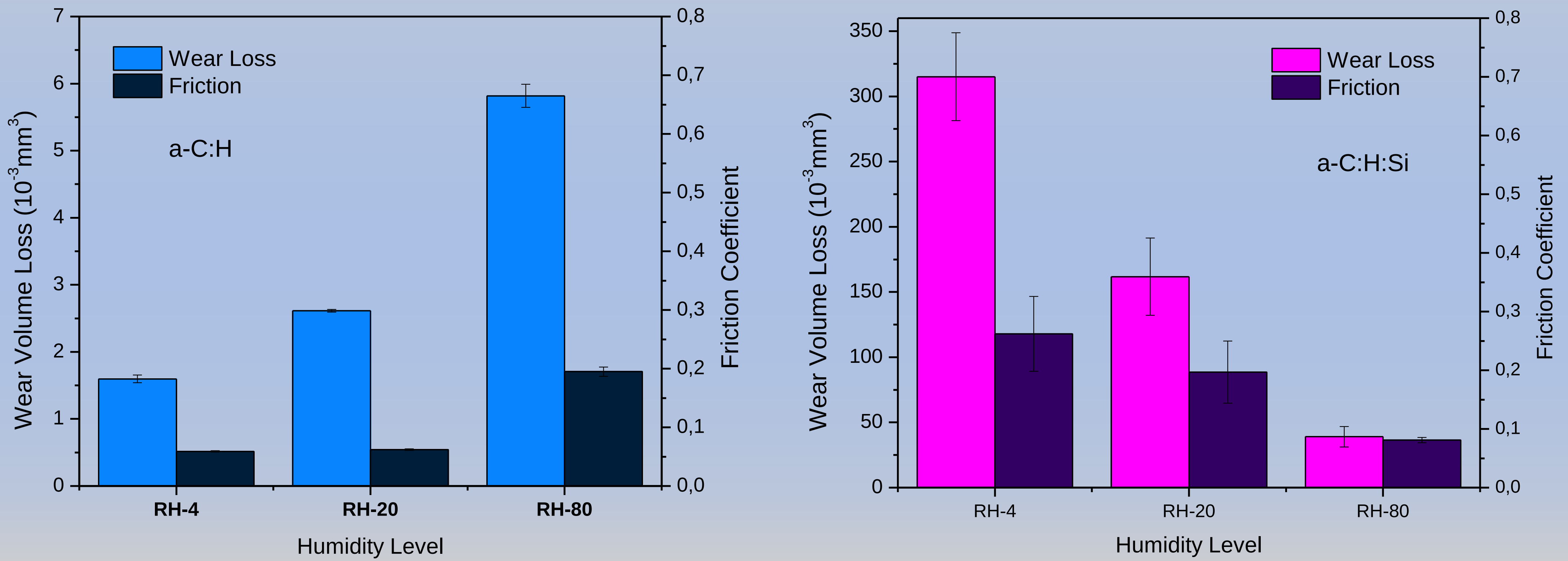


Si DLC

DLC 2 - Results - Tribology

Pin-on-Disk tests

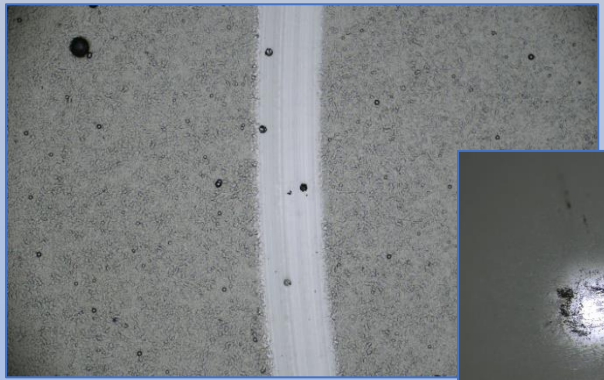
Influence of relative humidity on the wear loss

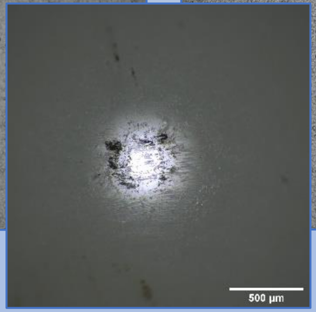


DLC 2 - Results - Tribology

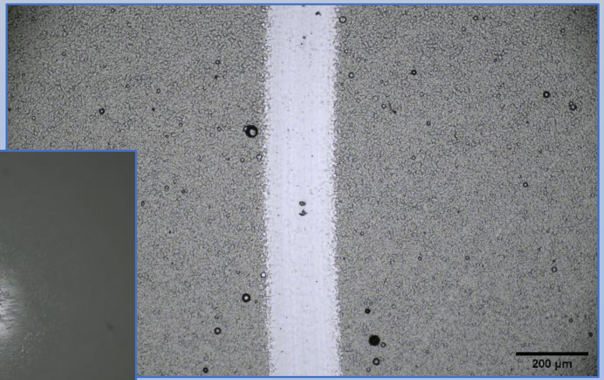
Pin-on-Disk tests

Impact of relatve humidity in the wear behaviour for a:C:H

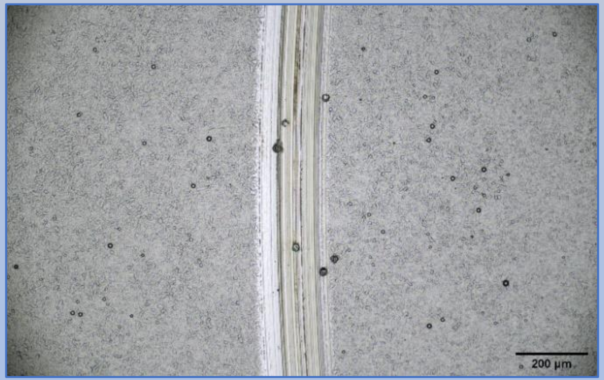




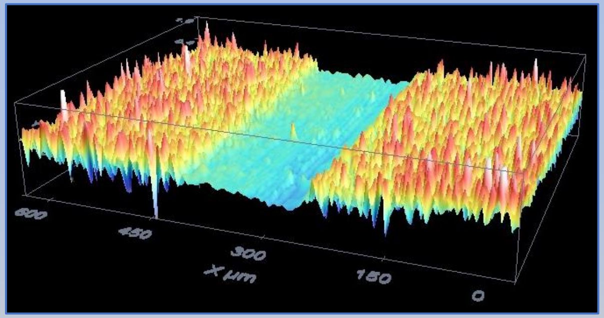
4% HR

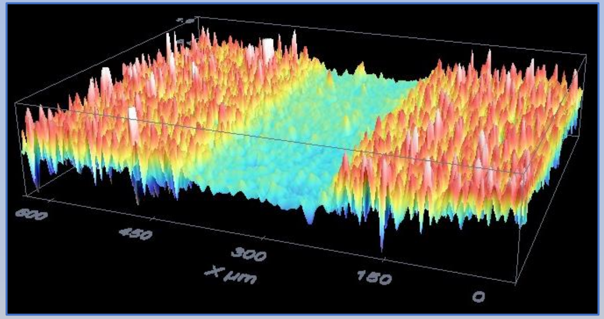


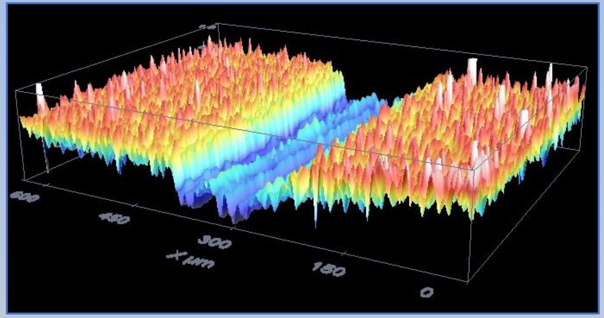
20% HR



80% HR



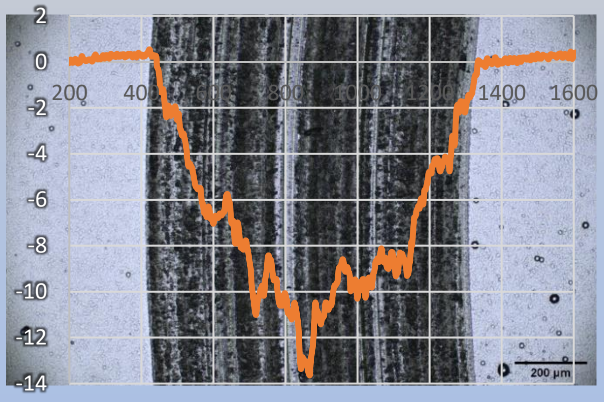




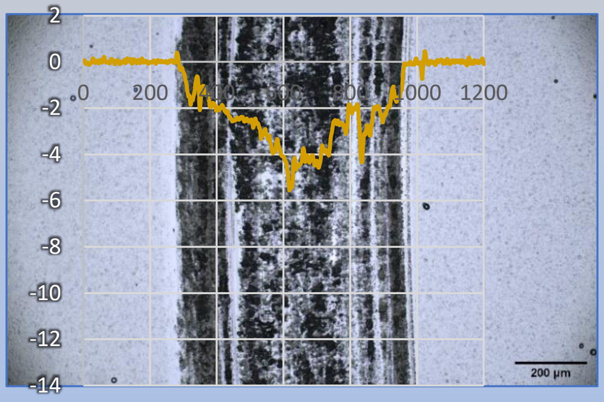
DLC 2 - Results - Tribology

Impact of relatve humidity in the wear behaviour for a:C:H:Si

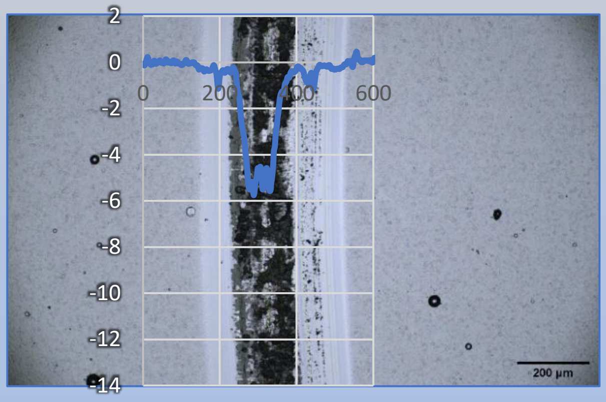
Pin-on-Disk Tests
Wear scars



4% HR

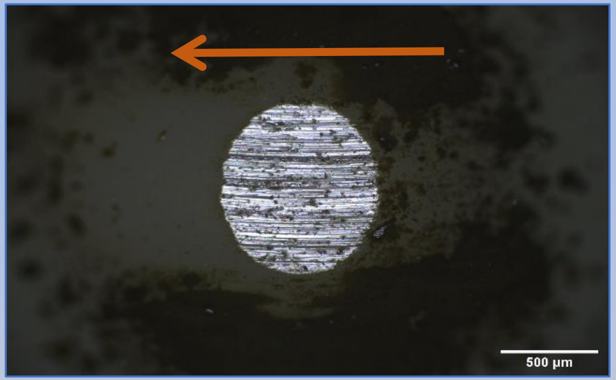


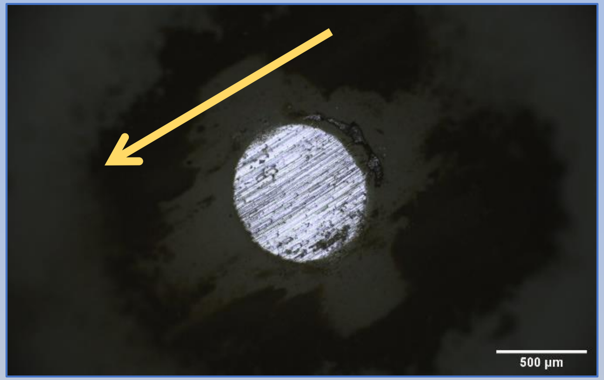
20% HR

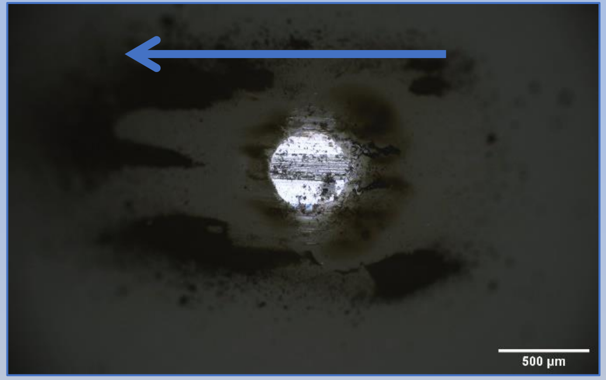


80% HR

Counterparts







DLC 2 - Partial Conclusions

Si addition

At Room Temp. and 20% Humidity

+ Hardness   + Fragility   + Disorder   ++ Wear loss   + Friction Coeff.

Increasing Humidity on Wear behaviour

Without Si	+	Wear loss
	+	CoF
With Si	−	Wear Loss
	−	CoF

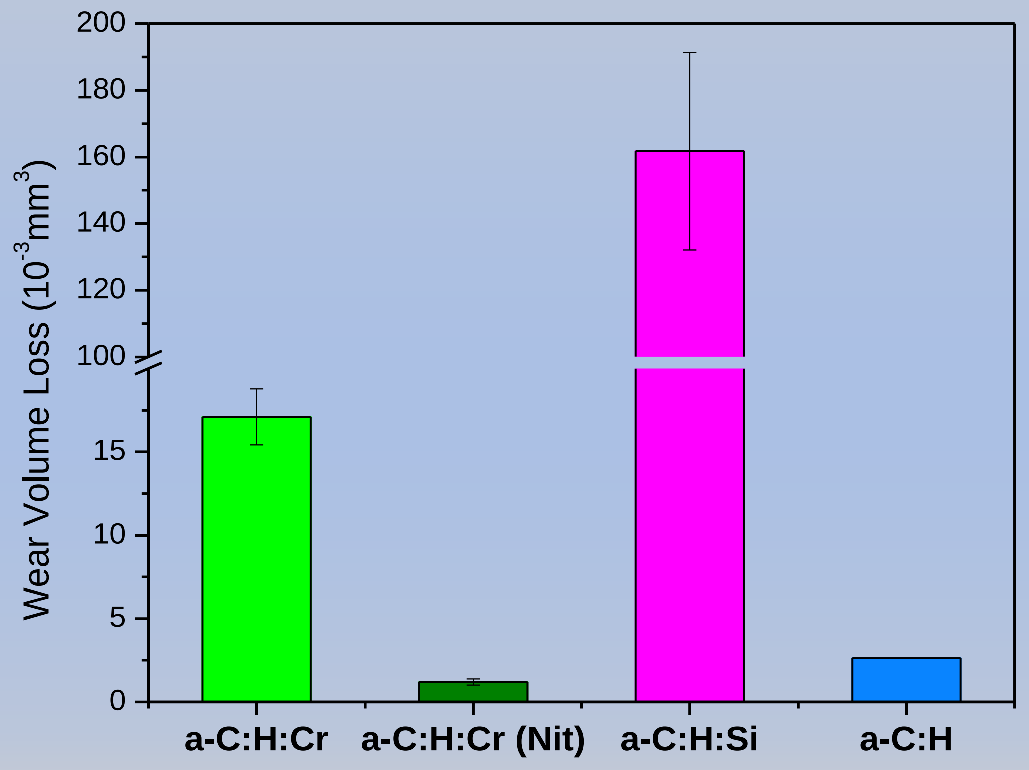
DIFFERENT TRIBOLAYERS

1) 80% HR - Silica sol debris layer
2) 4% HR - SiOx(OH)y gel

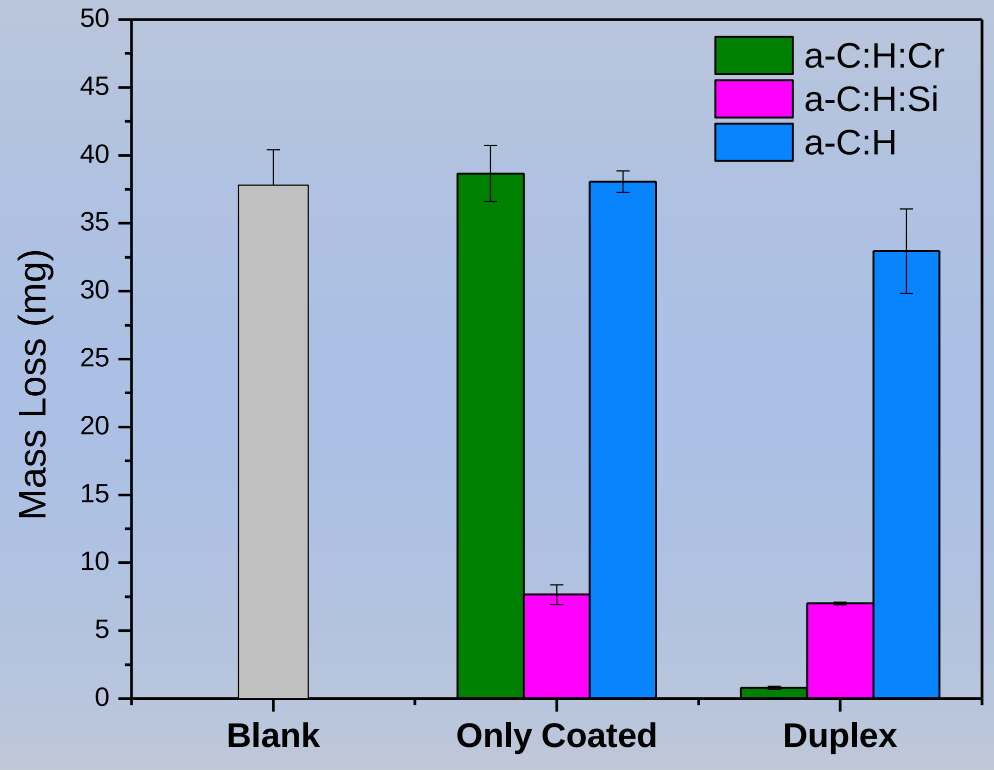
Lanigan et al., Tribology International 82 (2015) 431–442
Bai et al., Wear 484-485 (2021) 204046

All DLCs – Compared wear loss

Pin-on-Disk tests



Abrasive G65 Tests



Conclusions

- Nitriding as pre-treatment is necessary for thin films (under five microns) to withstand severe wear conditions.
- In the case of the soft and thick DLCs, the films can absorb enough energy to resist wear on its own.
- In pin on disk tests, increasing humidity in work conditions improves wear resistance and lowers CoF in Si containing DLCs but is the opposite in not doped DLC (different tribolayers).
- In abrasive wear tests, the Si-DLC is better than the Si free DLC.
- But in this test the best result was achieved by the multilayer Cr/CrN/DLC.

Aknowledgments

- ✓ To the financial support of the UTN and Faculty of Concepción del Uruguay
- ✓ To the Austrian Ernst Mach scholarship for Francisco Delfin
- ✓ The researchers of Surface Engineering Group (UTN) for the useful discussions
- ✓ To the students for the lab help
- ✓ To all of you for your kind attention!

Our social networks

@gis.utn

/GrupoGisUTN

Grupo de Ingeniería de Superficies

Grupo de Ingeniería de Superficies

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

Contact

Phone

+54 3442 - 425541 Ext. 130

Address

Universidad Tecnológica Nacional
Facultad Regional Concepción del Uruguay
Ingeniero Pereira 676
E3264BTD Concepción del Uruguay – Entre Ríos – Argentina

Mail

gis@frcu.utn.edu.ar
sonia@frcu.utn.edu.ar

Thanks!   Gracias!