MetAGEAR project activities – work package 3

Surface treatments and coatings for gears

M. Bononi, R. Giovanardi, L. Lusvarghi, T. Manfredini, P. Puddu, V. Testa
(Dipartimento di Ingegneria «Enzo Ferrari» e Centro InterMech - MO.RE.)

A. Ballestrazzi, E. Gualtieri, A. Rota, S. Valeri
(Dipartimento di Scienze Fisiche, Informatiche e Matematiche e Centro InterMech - MO.RE.)

University of Modena and Reggio Emilia

The Gear Day – 24th May 2018

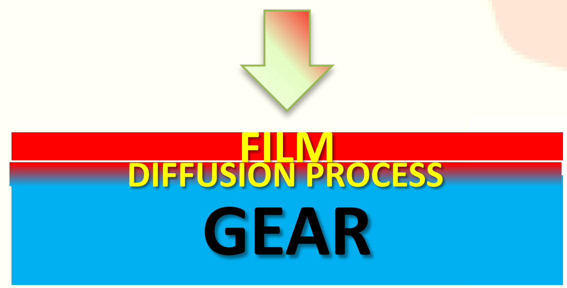
WORK PACKAGE 3 – MAIN ACTIVITIES

1) ***Development and optimization of thermochemical processes*** for the enhancement of the tribological performance

solid state diffusion ***into*** the material ➔ strengthening by solid solution and precipitation

2) ***Development and optimization of vapor deposited films (PVD, PECVD)*** for the enhancement of wear resistance and friction reduction

vapor diffusion of the coating material towards the material surface ➔ deposition of a solid film ***onto*** the component



morphological change

3) ***Development of surface patterning techniques*** for the enhancement of the tribological performance

WORK PACKAGE 3 – WORKING FLOW

Summing up, these are the samples which will be studied and tested in MetAGEAR project aiming at an increased performance.

Manufacturing of LABORATORY SAMPLES → Quenching & Tempering → **Thermochemical treatments**
- Carburizing
- Nitriding

Quenching & Tempering ↓ grinding < 0.4 micron ↓ *reference for comparison*

Thermochemical treatments ↓ **2 kinds of samples**

Thermochemical treatments ↓ grinding < 0.4 micron ↓

Coated gears
- WC/C-H – «a»
- WC/C-H – «b»

Morphologically modified gears
- Topography «a»
- Topography «b»

← ***Laboratory Tests on disks*** ← ***Gear test rig (WP 1)*** ←

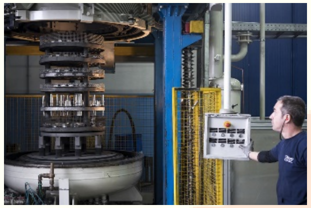
The winning combinations will be applied to COMMERCIAL GEARS to be tested in

Bonfiglioli
Forever Forward

Gear test rig (WP 1) ↓ *DATA INPUT FOR DATABASE (WP1)*

THERMOCHEMICAL TREATMENTS

LOW PRESSURE CARBURIZING



1) Heating at T > A_{c3} 920°C, 6 hours, in carburizing atmosphere
2) Cooling

The surface region affected by *carbon diffusion* undergoes martensitic trasformation → ***high hardness and compressive residual stresses***

STANDARD PROCESS FOR GEAR HARDENING

<u>Expected critical values</u>

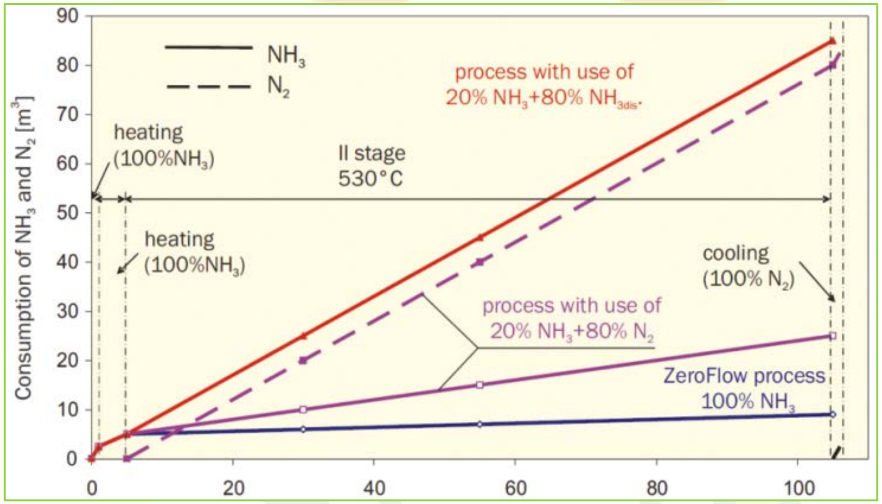
✓ Hardness - 60-62 HRC
✓ Case depth – 0.6-0.8mm

ZeroFlow® GAS *NITRIDING*

1) Heating at T < A_{c1} – 530°C, 36 hours
2) Cooling

Formation of iron nitrides (and nitrides of alloying elements, like Al, Cr, Mo) in the surface region affected by *nitrogen diffusion* → ***high hardness and compressive residual stresses***

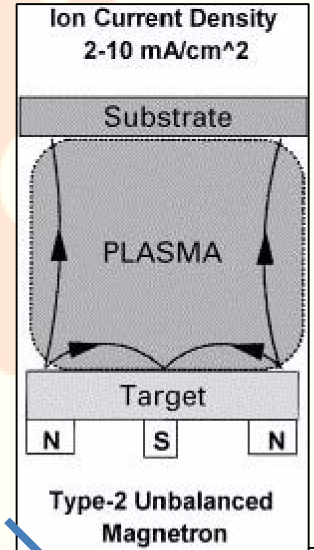
ALTERNATIVE ROUTE FOR GEAR HARDENING



FILM DEPOSITION PROCESSES

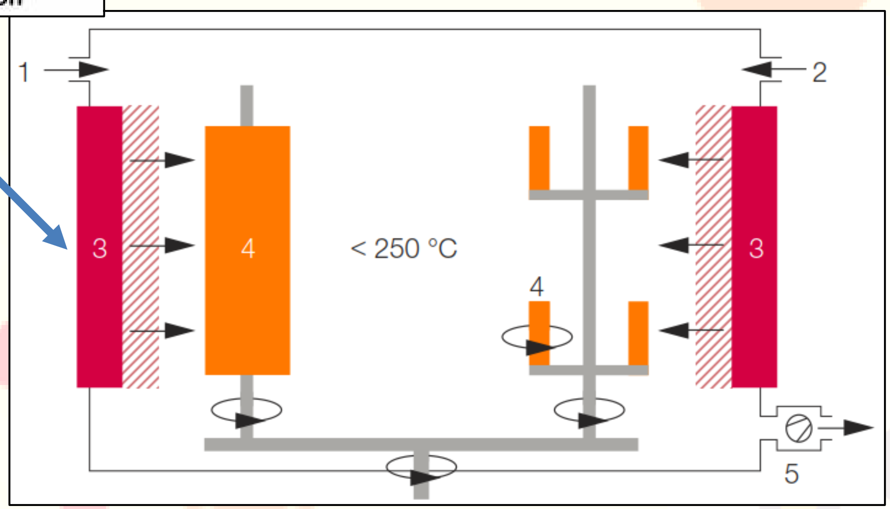
Following literature and existing gear applications (racing), the chosen solution had to be thin to respect tolerances, hard to withstand wear and behave as a solid lubricant to reduce friction, preventing scuffing.

DEPOSITION PROCESS – combined *magnetron sputtering*, a Physical Vapour Deposition (PVD) technique and Plasma Enhanced Chemical Vapour Deposition (PECVD)



batch process performed in vacuum

1. ***Argon (usually)***;
2. ***Reactive gas***;
3. planar magnetron evaporation source;
4. components to be coated;
5. vacuum pumps



- ✓ Argon, in plasma form, extracts vapors from the negatively charged solid target (3) = ***sputtering***
- ✓ A reactive gas is admitted to the chamber and is ionized/broken (es.: C_2H_2 => C_2H => CH…) = ***PECVD***

➔ *When they reach the component, they form a composite solid film and can react together*

From https://www.oerlikon.com/ecomaXL/files/balzers/oerlikon_HQ040EN_1011_web_locked.pdf&download=1

RESULTS

METAL DOPED DIAMOND-LIKE CARBON (different coating production lines)

FILM MATERIALS

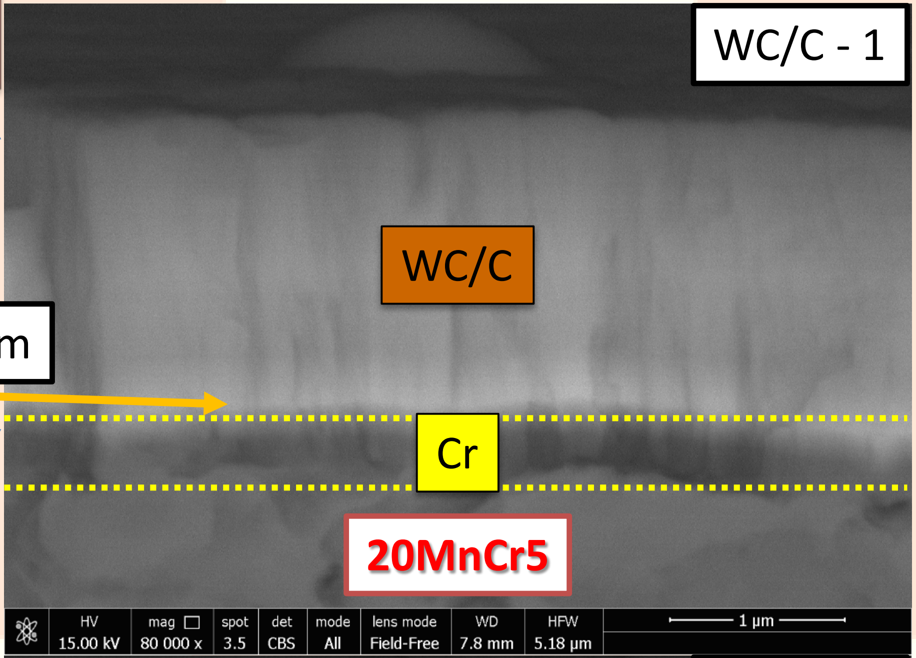
cross-sections fractured in liquid nitrogen

WC is active, C_2H_2 is admitted into chamber ➔ first WC vapours are in higher amounts, then C-based vapours become dominant => ***the surface is C rich***

2 µm

WC target becomes gradually active, Cr target gradually off

Cr target is active - magnetron sputtering



WC is active, C_2H_2 is admitted into chamber ➔ first WC vapours are in higher amounts, then C-based vapours become dominant => ***the surface is C rich***

2 µm

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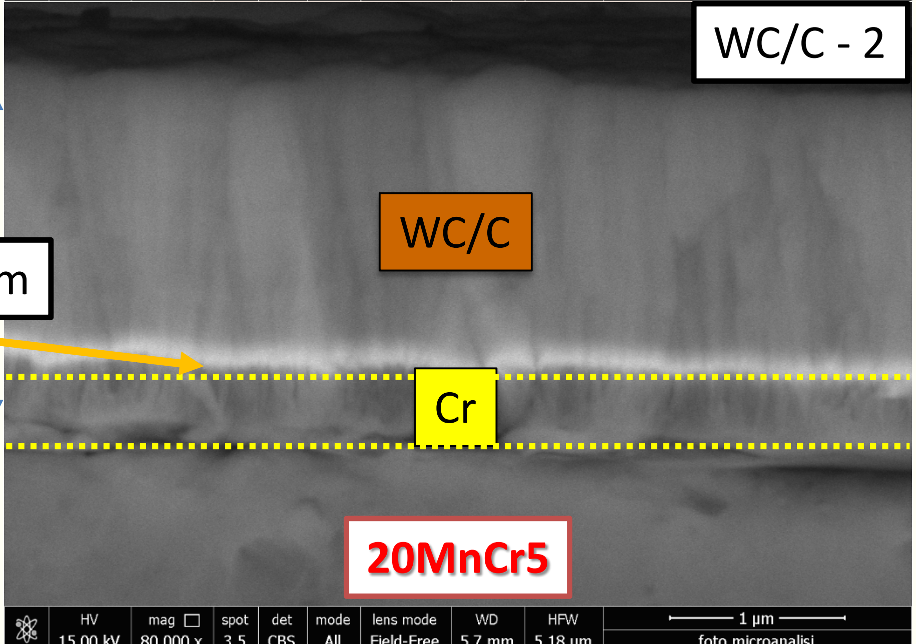
Cr target is active - magnetron sputtering



deposited at T < 200°C

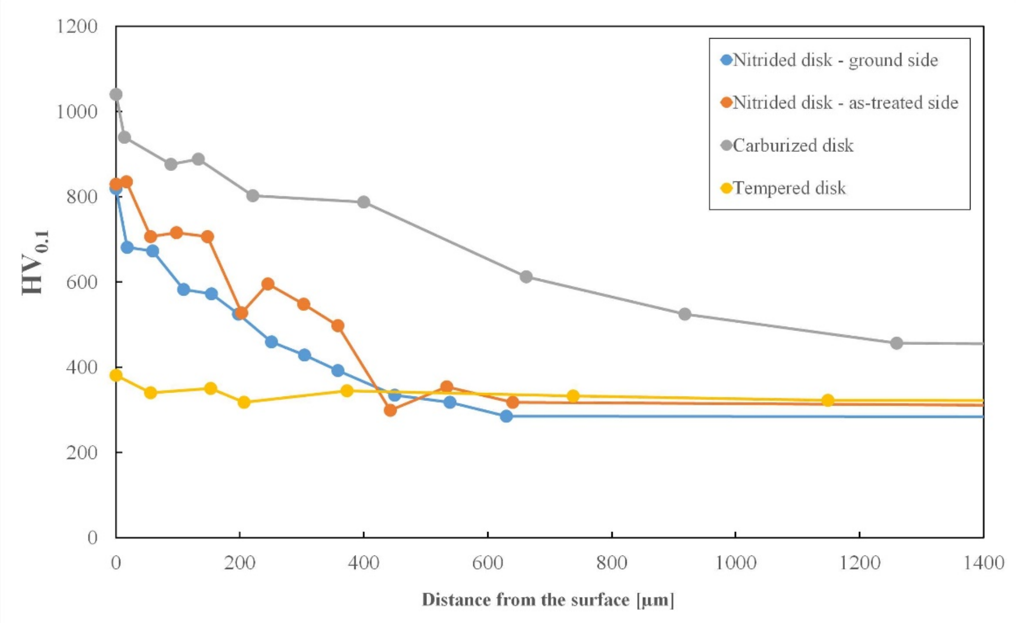
QUALITY CHECK: HARDNESS AND ROUGHNESS

✔ **Roughness < 0.4 µm**

Roughness → / ↓ Treatment/Film	Ra (micron)	Rz (micron)
Tempered	0.09	3.23
Carburized	0.09	2.46
Nitrided	0.07	2.22
T + WC/C-1	0.10	5.13
C + WC/C-1	0.07	2.19
N + WC/C-1	0.07	3.24
T + WC/C-2	*0.29*	4.83
C + WC/C-2	0.10	5.34
N + WC/C-2	0.10	4.09

Is still the high hardness due to case hardening present after grinding ?

Nitriding did not induce the highest hardness, but a significant surface hardness increase is still present. ✔



Is the coating hardness within the range suggested for «metal-doped carbon-based» films for tribological applications ?

Film	Hardness (H_{IT} – HV)
WC/C – 1	10.2 GPa ± 2.5
WC/C - 2	*7.9* GPa ± 1.2

In WC/C commercial films, the hardness range is quite large due to:

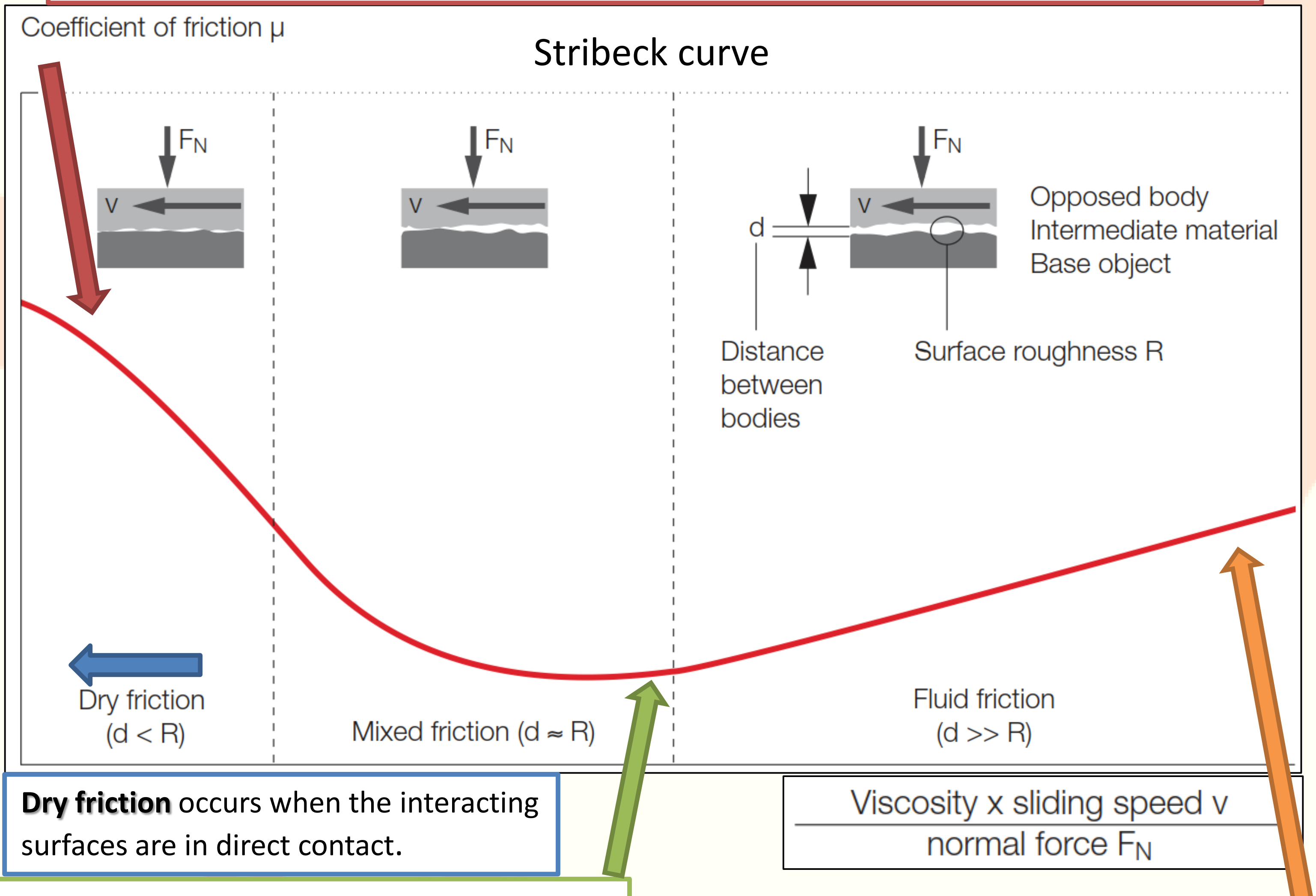
- ✓ film deposition parameters
- ✓ hardness tester equipment

✔

H_{IT} ~ ***8-12*** or 12-15 GPa
from Oerlikon Balzers website, Balinit C

TRIBOLOGICAL TEST – PARAMETER SELECTION - OVERVIEW

Boundary friction is an intermediate state between dry and mixed lubrication, in which adsorbed lubricant molecules cover the interacting surfaces.



Dry friction occurs when the interacting surfaces are in direct contact.

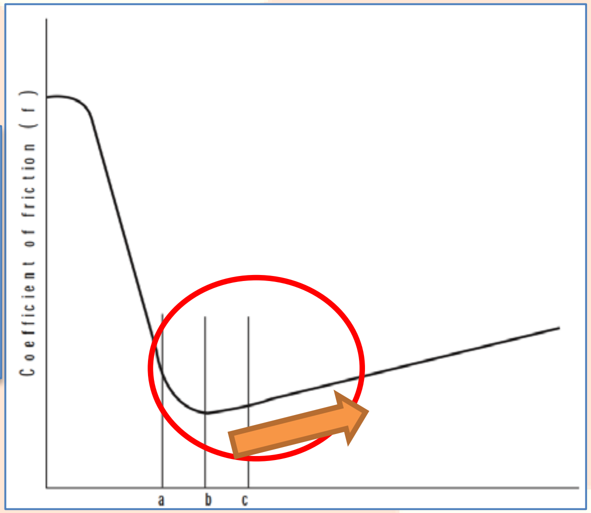
Mixed friction is a superposition of frictional states such as dry, boundary and fluid. Here the load is taken up partly by solid-to-solid contact and partly by a load-bearing lubricant film.

Fluid friction is friction in a liquid lubricant film that completely separates one contact partner from the other (hydrodynamic and hydrostatic friction).

TRIBOLOGICAL TEST – PARAMETER SELECTION – WHY ?

1) The first goal of the WC/C films is to decrease friction in poor lubrication conditions (they should act mainly on the sliding component in the gear motion)

➔ ***improvement of gear efficiency and scuffing prevention ?***



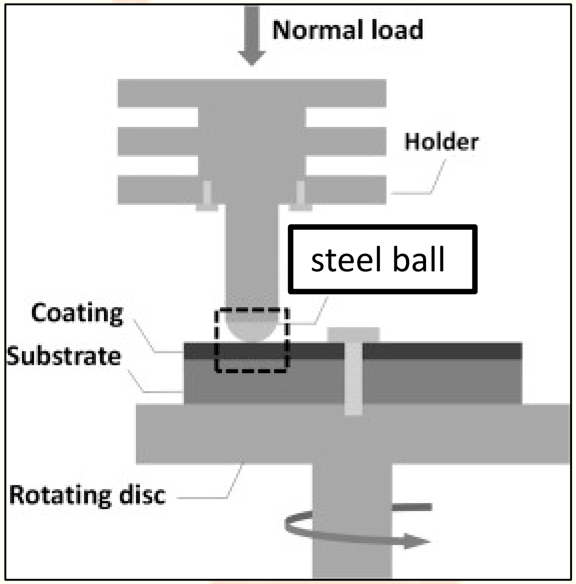
2) The mixed lubrication zone is where the lower friction occurs. It is often the range where many mechanical components would maximize efficiency. At the same time, a reasonable factor of safety shifts design requirements ***to the left*** of the Stribeck curve.

3) ***The tribological conditions*** must be reasonably ***close*** to the gear contact conditions, ***where/when sliding conditions occurs***

➔ ***load and speed*** must be of the same order of magnitude, ***the lubricants*** suggested for gears

- ✓ Load = 15 N => ***Hertzian pressure = 0.87 GPa***
- ✓ Speed = ***up to 1.5 m/s***
- ✓ Lubricants = 80 ml
  1) ***Oil ISO 68*** – used as lubricant in the gear test rig – ***68 cSt @ 40°C*** / ~ 10 cSt @ 100°C
  2) ***Oil ISO 150*** – standard lubricant for gear applications – 150 cSt @ 40°C /***20 cSt @ 100°C***

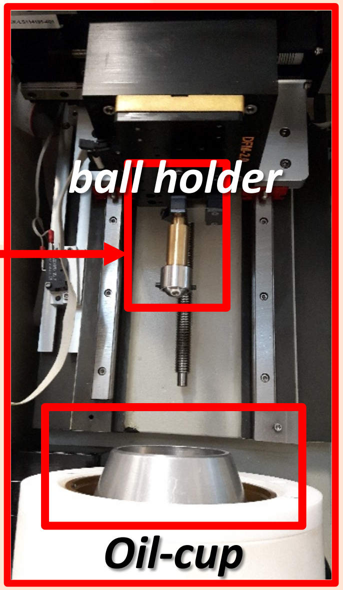
TRIBOLOGICAL TEST – PARAMETER SELECTION – HOW ?



pin-on-disk set up

ASTM G99

ISO 18535:2016
(applied to Diamond-like carbon film)

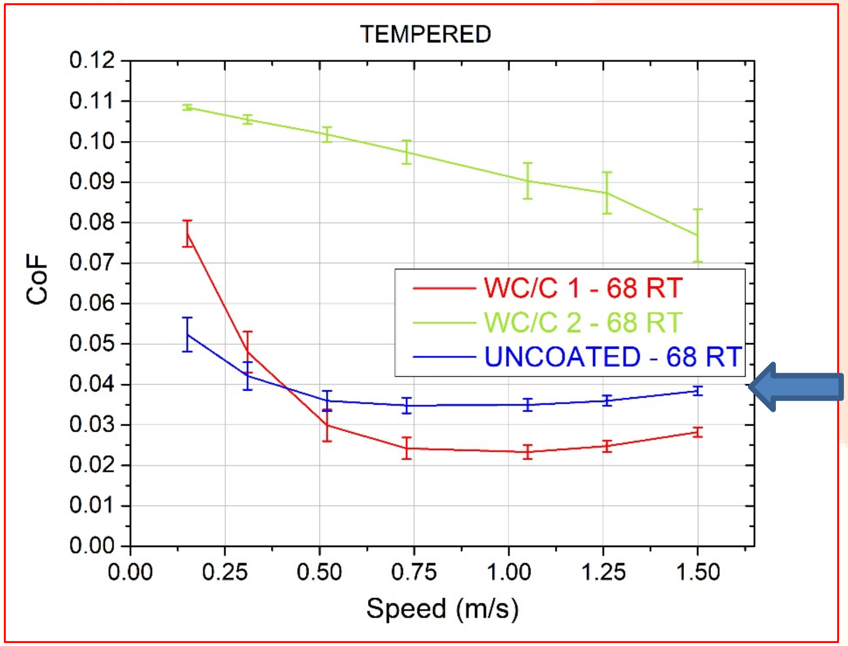


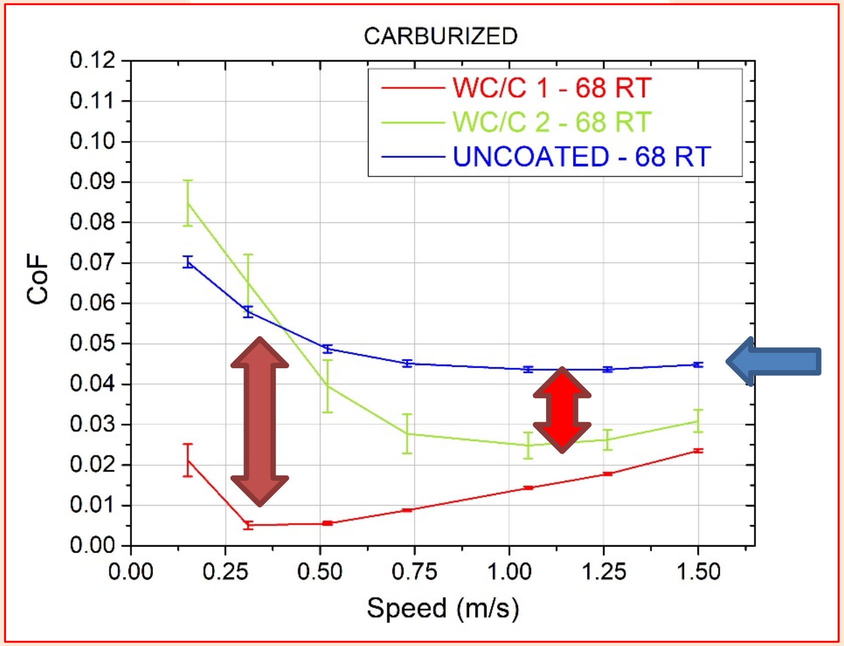
The tribological tests have been planned to a Stribeck curve, ***varying the sliding speed by steps***, *keeping the load constant* in the *same oil bath*.

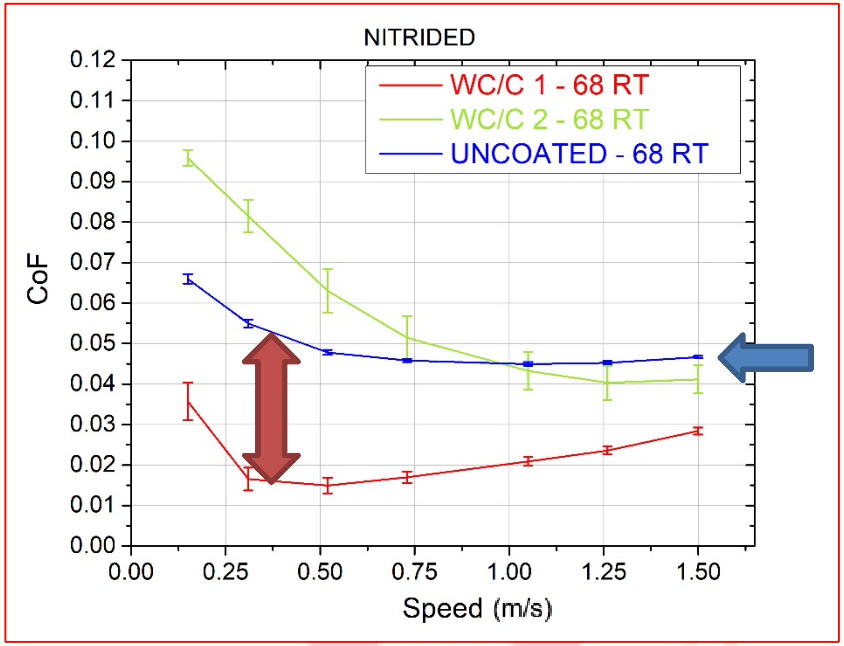
- ✓ *A 100Cr6 hard steel ball* (diameter 15 mm) has been chosen as static counterpart
- ✓ *2 ascending-descending speed ramps* have been carried out for each sample to obtain *4 repetitions for each point* of the speed/friction curve in order to collect an exhausitive statistical data pattern. *Friction coefficient value for each step is the average of the 4 measurements*.
  Variation of the rotational speed: 75, 150, 250, 350, 500, 600, 750 rpm; 60 seconds for each step.
- ✓ *Tests have been performed at RT (25°C – Oil 68 η = 84 cSt) and HT (80°C – Oil 150 η = 29 cSt)*:
  - Oil cup was equipped with a thermocouple placed near the pin/disk contact area
  - Thermal evolution of the oil bath has been monitored during each test

RESULTS

TRIBOLOGICAL BEHAVIOUR – Synthetic Oil 68 RT





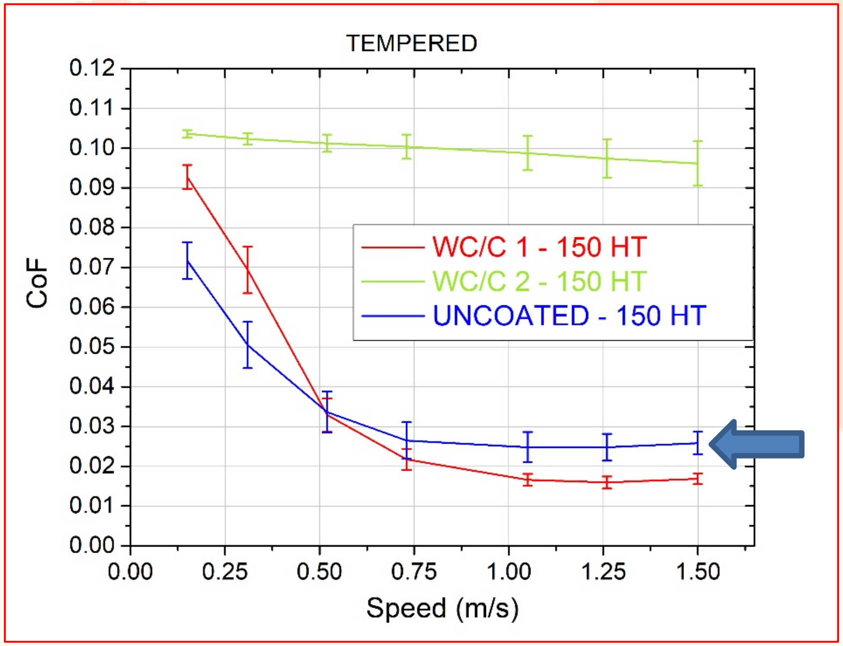


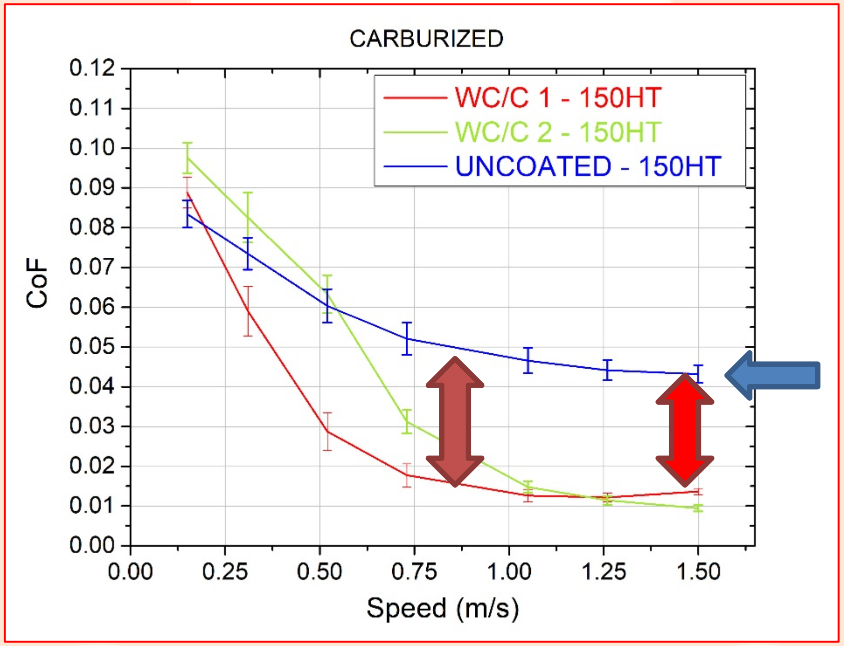
- ✓ *There is no great difference in CoF due **only** to the different thermochemical* treatment along the whole speed range

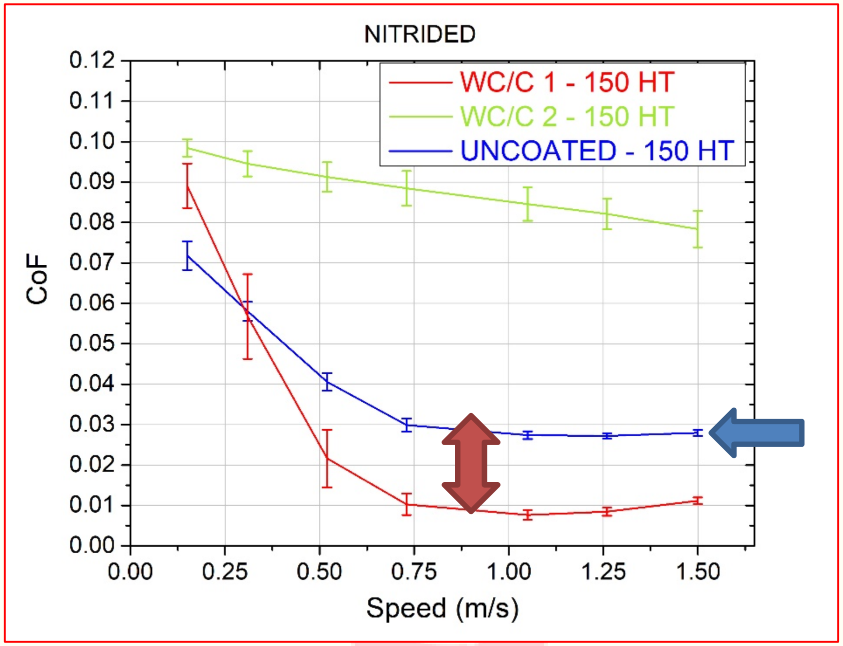
- ✓ **The coated samples behave quite differently** ***WC/C 1*** *can decrease CoF down to 3-4 times, especially in the worst lubrication conditions (low speed) AND with high hardness substrate (carburized best); WC/C – 2 is beneficial only on carburized samples.*

RESULTS

TRIBOLOGICAL BEHAVIOUR – Synthetic Oil 150 HT



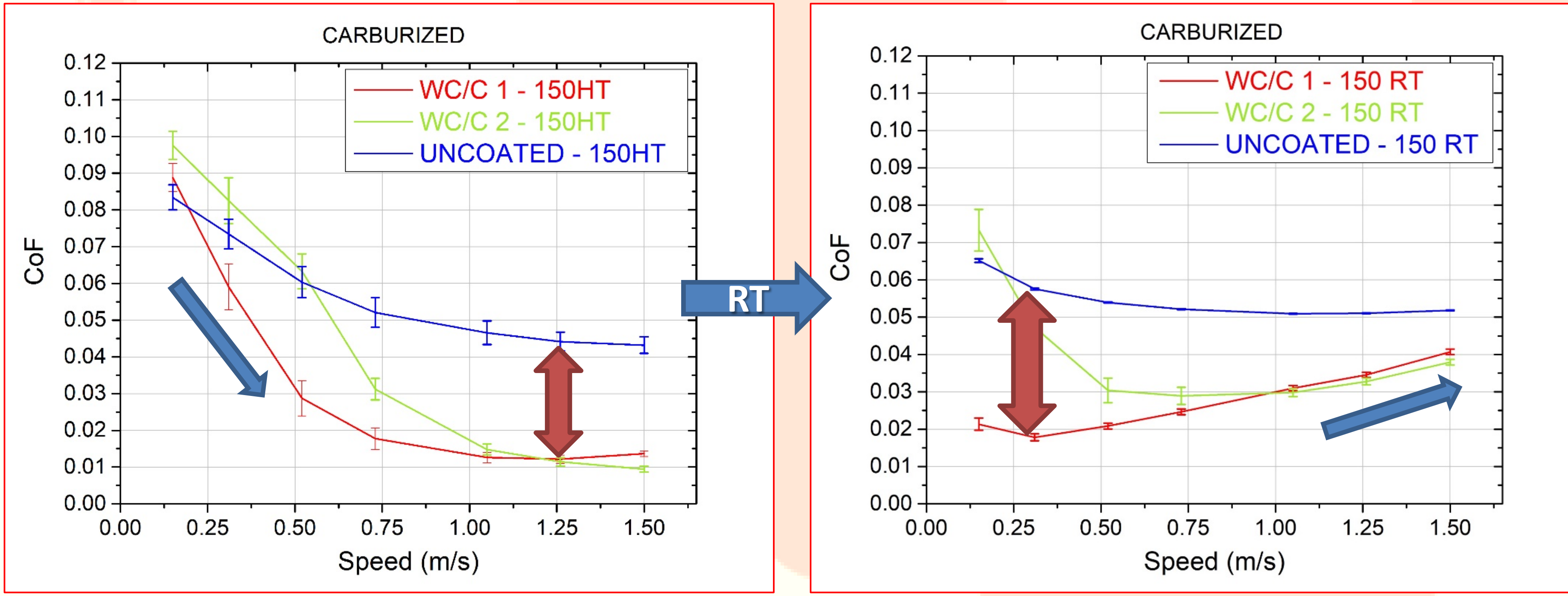




- ✓ *There is no great difference in CoF due* ***only*** *to the different thermochemical, with tempered and nitrided samples behaving best (0.07 → 0.03) than carburized (0.08 → 0.04)*

- ✓ **The coated samples behave even more differently.** ***WC/C 1*** *can decrease CoF down to 3-4 times, increasing speed AND with high hardness substrate (carburized best);* ***WC/C – 2 is detrimental*** *on both tempered and nitrided sample, beneficial only in the carburized case.*

TRIBOLOGICAL BEHAVIOUR – Synthetic Oil 150 RT & HT



80°C, η = 29 cSt

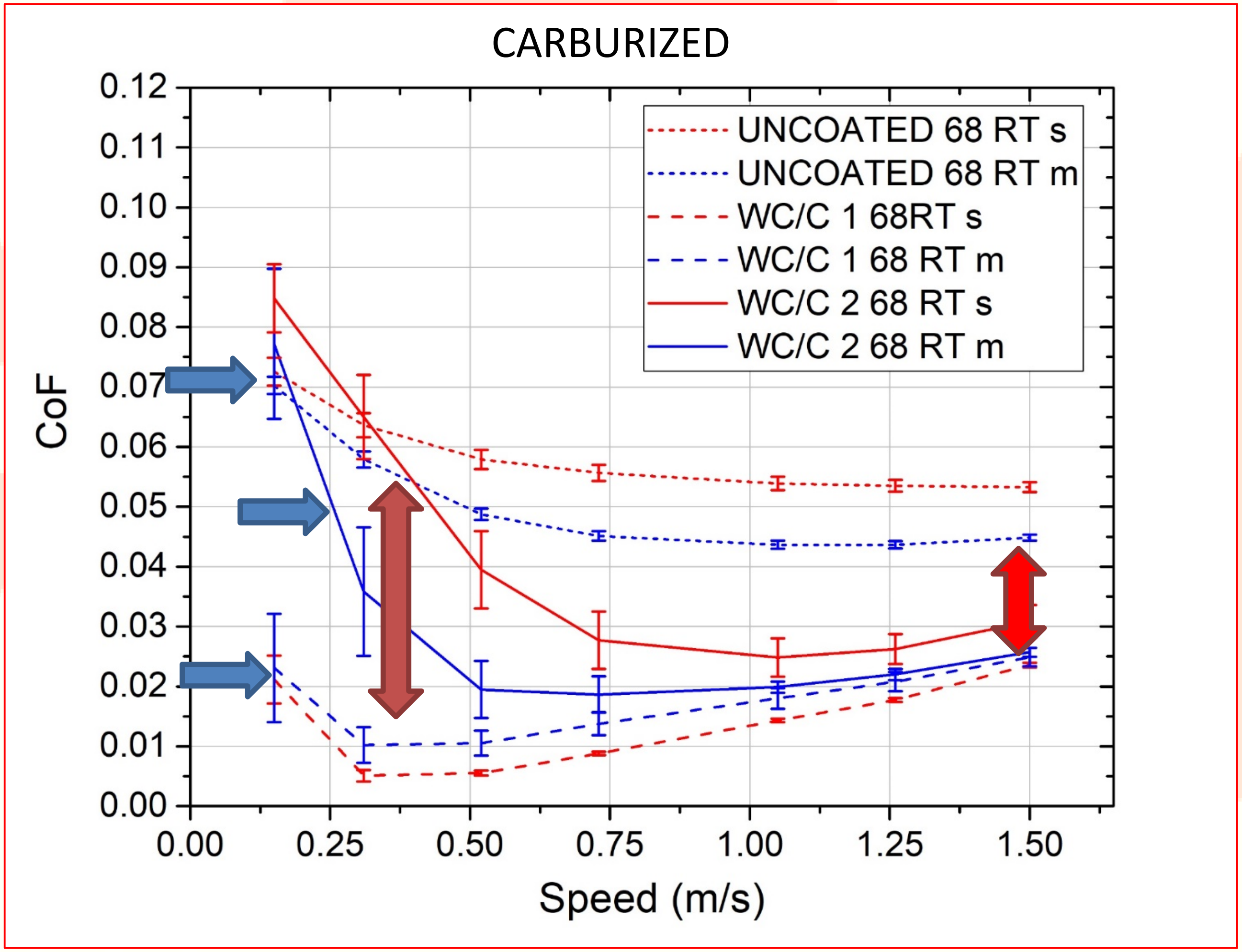
30°C, η = 200 cSt

- ✓ *At RT*, ↑ higher viscosity, **CoF are increasing at high speed** (moving from the mixed to the lubrication friction zone of the Stribeck curves); *at HT*, ↓ lower viscosity, **CoF are still decreasing with speed** (entering the mixed zone).

- ✓ **Both WC/C reduce friction**, with *WC-C showing the best performance at lower-medium speeds at RT* and *higher speeds at HT;* **at RT**, **at higher speeds**, **CoF increase** going towards uncoated sample values, since the solid lubrication is no more effective (fluid friction activated).

RESULTS

TRIBOLOGICAL BEHAVIOUR – Oil 68 RT synthetic and mineral oil

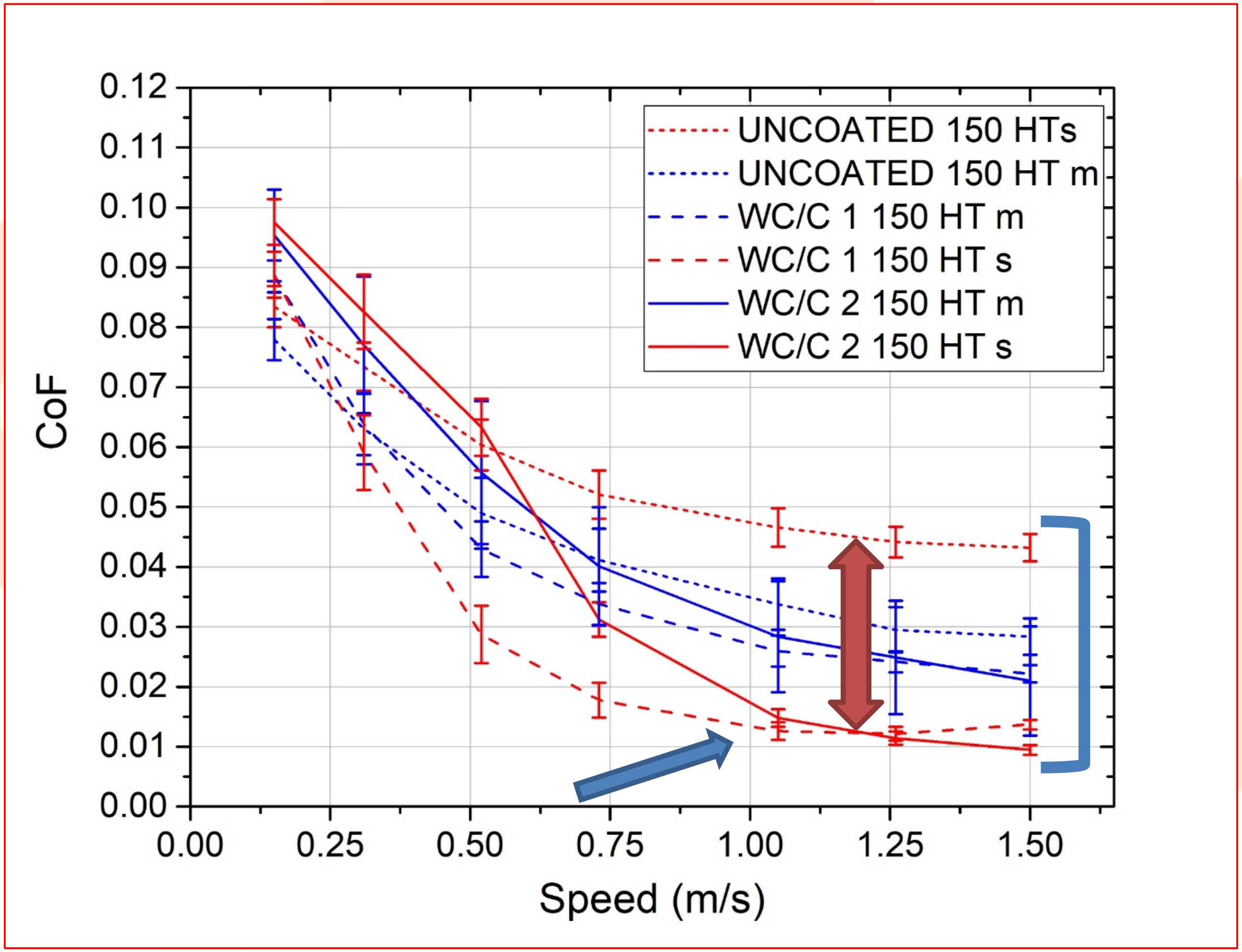


✓ *At RT*, **mineral oils systematically show a lower or similar CoF** (blue lines)

✓ **Both WC/C decrease CoF**, *WC/C 1 performs best at lower speed where mixed friction is still very active*

RESULTS

TRIBOLOGICAL BEHAVIOUR – Oil 150 HT synthetic and mineral



- ✓ *At HT*, **the two kinds of oils show a narrower range of CoF,** *with coated samples reaching lowest CoF with synthetic oil*

- ✓ *At HT*, **WC/C 1 decreases friction in both oils,** *with the best performance in synthetic oil and higher speed (still in mixed friction mode).*

FILMS RELIABILITY/ADHESION

WC/C 2 on nitrided sample

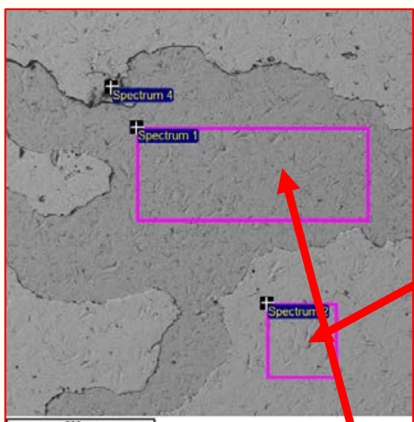
Optical inspection suggests severe delamination of the film

WEAR TRACK ANALYSIS

- ✓ WC/C 1 – no delamination
- ✓ **WC/C 2 – delamination:**
  - *nitrided - oil 68 RT*
  - *nitrided - oil 150 HT*
  - *carburized - oil 68 RT*

WC/C films are multilayers. Where is the problem ?
Cohesion among layers or adhesion of the whole film ?

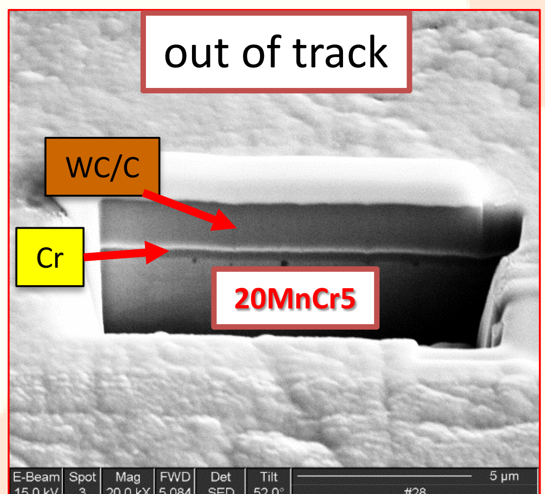
SEM/EDS – surface microanalysis

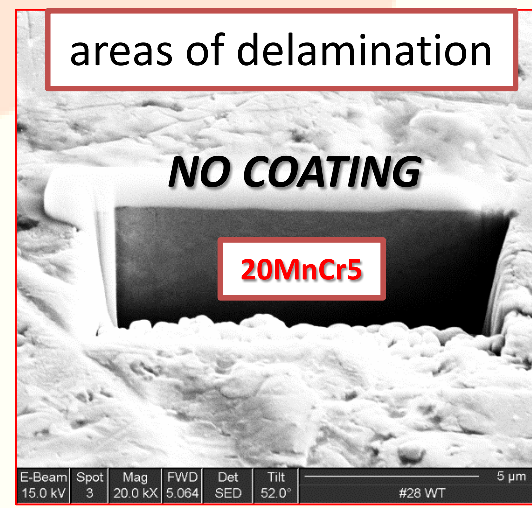


FILM
W, C, Cr

areas of delamination:
Fe, Cr, Mn, Si – no W

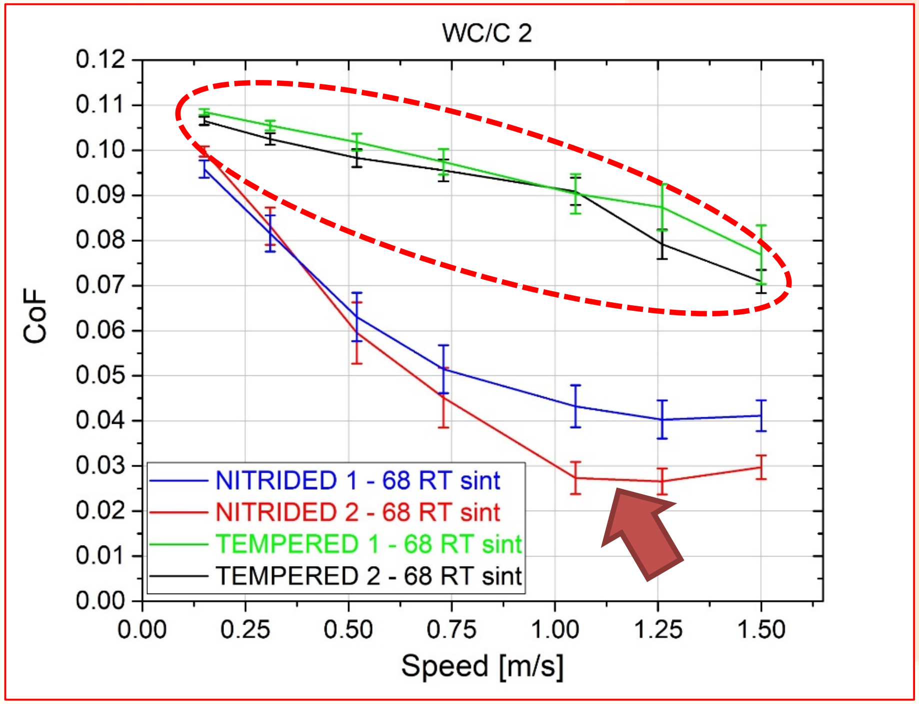
FIB/SEM – surface analysis





ADHESION

FILMS RELIABILITY/ADHESION



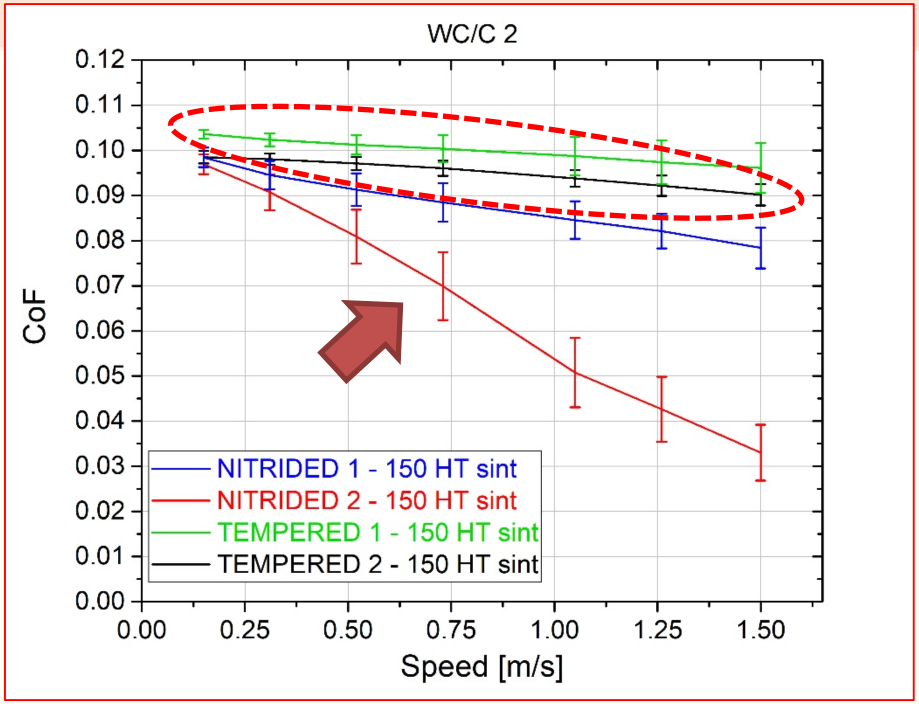
Further tests have been performed on several samples *to investigate WC/C 2 higher friction and delamination behavior*

The ***higher friction*** of WC/C 2 on tempered samples is **confirmed** in both oil and temperatures conditions

The ***delamination*** on nitrided samples is **not confirmed** and the measured friction is ***lower*** at all speed (still ***higher*** than WC/C 1)

wear track

WC/C 2 on nitrided 2 sample

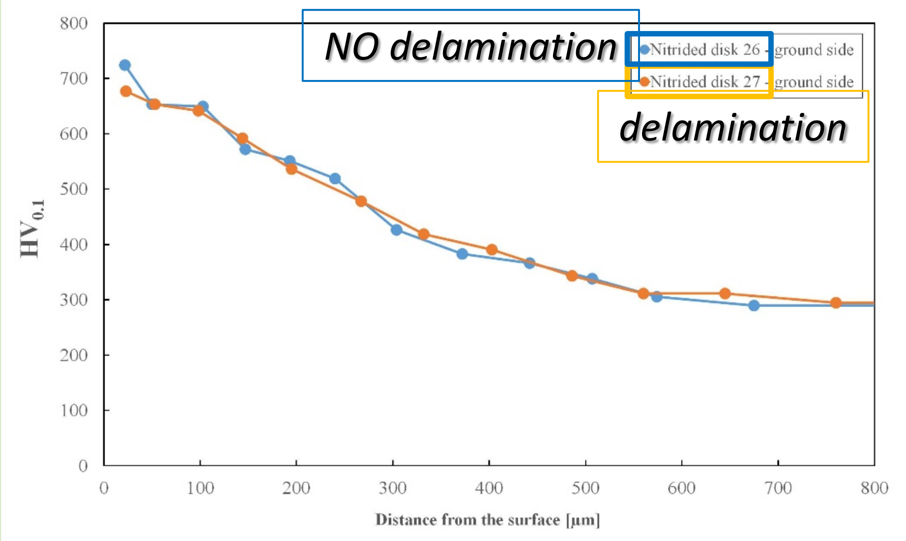


ADHESION due to an unreliable process ?

FILMS RELIABILITY/ADHESION

Can be an unreliable thermochemical process be the cause of adhesion loss ?

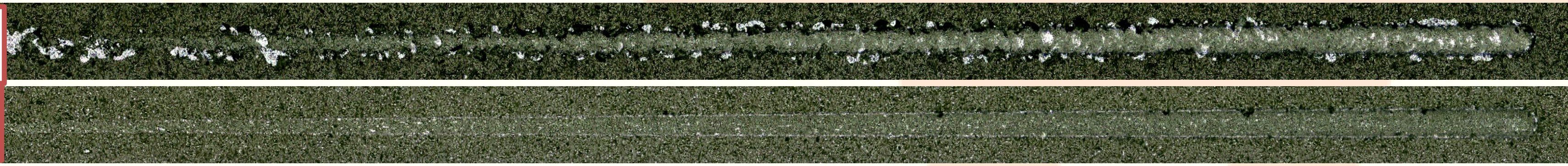
There is no difference in the hardness profile between samples with the same thermochemical treatment

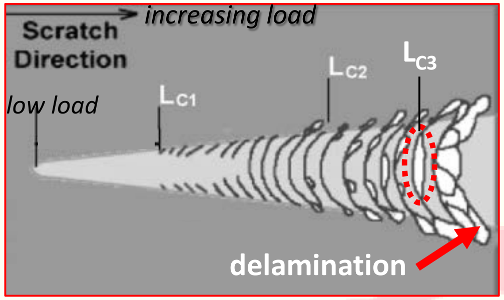


Can be an unreliable sputtering deposition process be the cause of adhesion loss ?

«TRIBO» **DELAMINATION**

NO «TRIBO» DELAMINATION





scratch test – 200µm according to ISO20502

	Load (N) → Sample ↓	L_{C1}	L_{C2}	L_{C3}	delamination	«TRIBO» behavior
1	Temp - WC/C - 1	6.9	20.5	22.74	/	
1	Carb – WC/C – 1	15.41	/	/	/	
1	Nitr – WC/C – 1	9.9	20.2	22.9	/	
2	Temp – WC/C - 2	n.m.	7.1	8.4	19	*high friction*
2	Carb – WC/C – 2	n.m.	6.8	6.8	/	*DELAMINATION*
2	Carb – WC/C - 2	n.m.	18	22.3	/	
2	Nitr – WC/C – 2	n.m.	3.6	4.1	/	*DELAMINATION*
2	Nitr – WC/C 2	n.m.	14.1	16.5	/	

CONCLUSIONS

- ✓ *Carburizing and nitriding have been selected to harden both disks and gears*; tempered samples have been prepared as «reference».

- ✓ From literature and market solutions focused on gear applications, *«carbon-based» films (metal-doped DLC, or «WC/C»)* have been selected to coat hardened samples
- ✓ *2 WC/C films have been deposited by magnetron sputtering and PECVD vapor deposition process* in different production lines. A Cr adhesion layer should improve the adhesion of a carbon rich layer, which should provide low friction properties.

- ✓ Pin-on-disk experiments have been carried out *to «simulate» the sliding conditions in a gear contact* choosing resonably close load, speed and kind of lubrication.

- ✓ *There is not a major advantage in friction reduction due to the thermochemical process*
- ✓ <u>IF the film shows good adhesion (WC/C 1)</u>, **then a significant friction reduction has always been detected on *carburized* and nitrided coated samples, highest in the mixed lubrication zone where the CoF minimum is measured** according to tribological conditions (speed, oil viscosity).
- ✓ <u>IF the film does not show a reliable good adhesion (WC/C 2)</u>, **then the friction reduction is more limited and resistance to delamination not predictable.**

WORK IN PROGRESS & FUTURE DEVELOPMENT

- ✓ Experimental: **«endurance» pin-on-disk tests** will be performed increasing the load up to 2GPa and test duration up to hours to check where WC/C 1 delamination limit occurs.
- ✓ Experimental: **«superfinishing»**, i.e. *roughness reduction < 0.1 AND R_z abatement*, will be applied to the thermochemically treated samples and, then, tested with and without WC/C 1 coating
- ✓ To be understood: *the connections between the lubricated sliding tests on disks and the tests on gears.* Are there conditions when the results can be «directly» translated from disks to gears ?

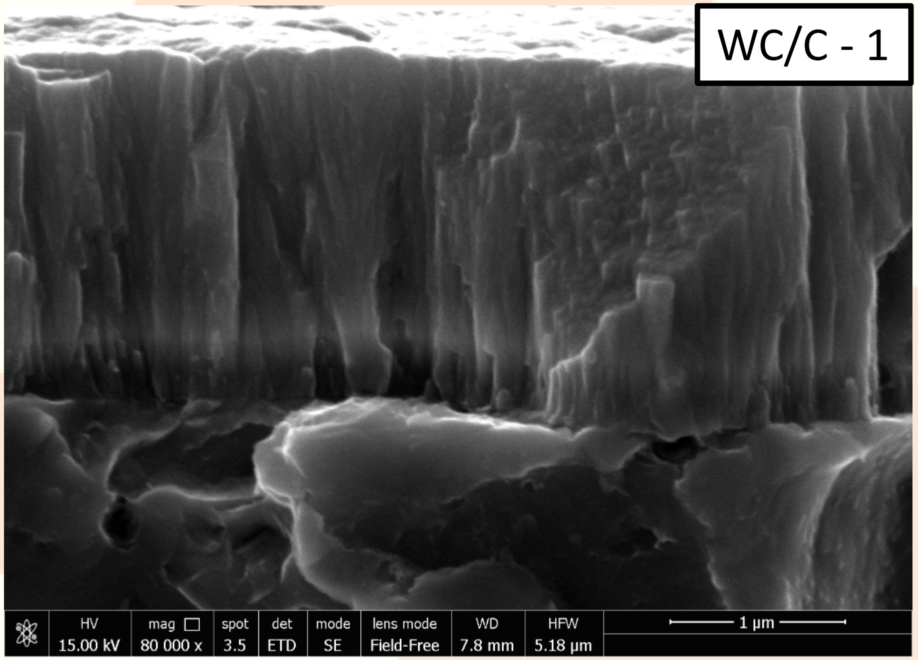
- ✓ Experimental, *surface patterning*: **disks and gears have been patterned by laser treatment.** Both pin-on-disk tribometer tests (disks) and test rig campaign (gears) are running.

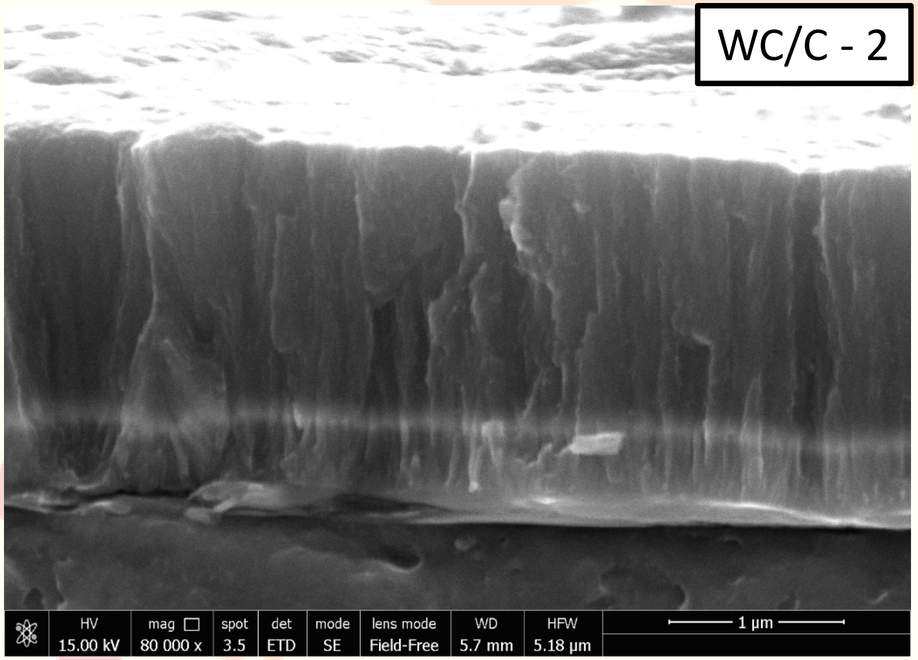
→ *New projects which systematically investigate* the role of thermochemical treatments and thin films on the possible improvement of gear applications for different gears and loading/speed/lubrication condition

Thank you for your kind attention!

luca.lusvarghi@unimore.it
roberto.giovanardi@unimore.it

enrico.gualtieri@unimore.it


WC/C - 1
HV 15.00 kV
mag 80 000 x
spot 3.5
det ETD
mode SE
lens mode Field-Free
WD 7.8 mm
HFW 5.18 µm
1 µm


WC/C - 2
HV 15.00 kV
mag 80 000 x
spot 3.5
det ETD
mode SE
lens mode Field-Free
WD 5.7 mm
HFW 5.18 µm
1 µm

Hydraulic oil ISO 68				
Mineral based hydraulic oil				
Property	**Value in metric unit**		**Value in US unit**	
Density at 60°F (15.6°C)	0.880 *10^3	kg/m³	54.9	lb/ft³
Kinematic viscosity at 104°F (40°C)	68.0	cSt	68.0	cSt
Kinematic viscosity at 212°F (100°C)	10.2	cSt	10.2	cSt
Viscosity index	135		135	
Flash point	204	°C	400	°F
Pour Point	-40	°C	-40	°F
Aniline Point	88	°C	190	°F
Color	max.7.0		max.7.0	

Hydraulic oil ISO 150				
Mineral based hydraulic oil				
Property	**Value in metric unit**		**Value in US unit**	
Density at 60°F (15.6°C)	0.882 *10^3	kg/m³	55.1	lb/ft³
Kinematic viscosity at 104°F (40°C)	147	cSt	147	cSt
Kinematic viscosity at 212°F (100°C)	14.6	cSt	14.6	cSt
Viscosity index	98		98	
Flash point	286	°C	547	°F
Pour Point	-24	°C	-11	°F
Aniline Point	119	°C	246	°F
Color	max. 3.5		max. 3.5	