



Heat treatment conference  
and exhibition 2024

## NEW GENERATION OF POST-OXIDIZED BRAKE ROTORS

**NITREX SMART-ONC<sup>®</sup> – TACKLING CORROSION**

Saadia Nousir, [Marcin Stoklosa](#), Karl-Michael Winter



November 6/7, 2024

**NITREX**

# MOTIVATION



<https://rimsa.com/news/how-limitation-on-brake-emissions-by-euro-7-may-affect-the-friction-industry>

New Euro 7/VII emission standard

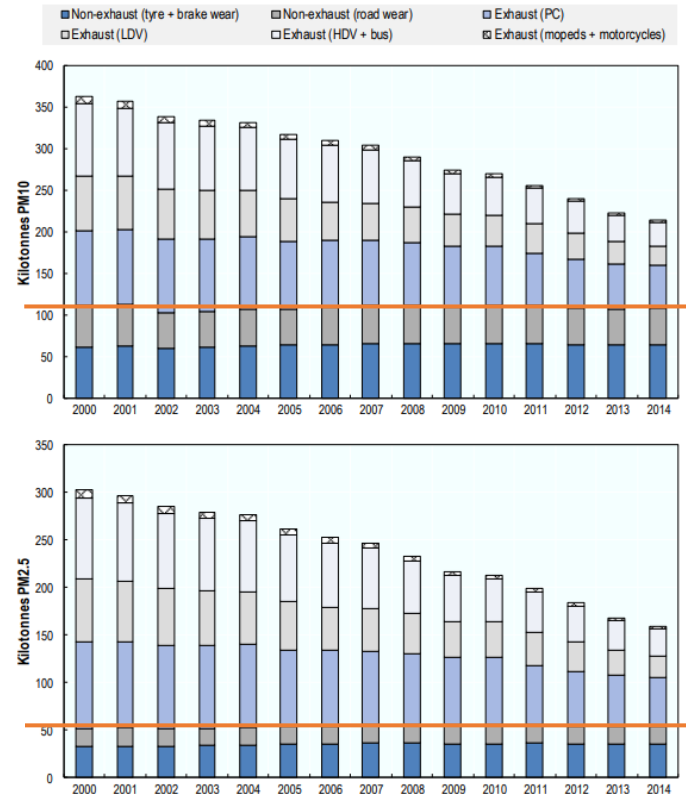
- effective as of 2025

# Euro 7/VII Emission Standards

With the continuous reduction of PM emissions from the exhaust (above orange line) while having constant emissions from non-exhaust (below orange line), the ratio of **non-exhaust PM** on total emissions is rising.

On the way to zero emissions, the new regulation is now **taking this into account**.

Figures taken from OECD report ENV/EPOC/WPIEEP(2020)4/FINAL on Non-exhaust emissions from road transport



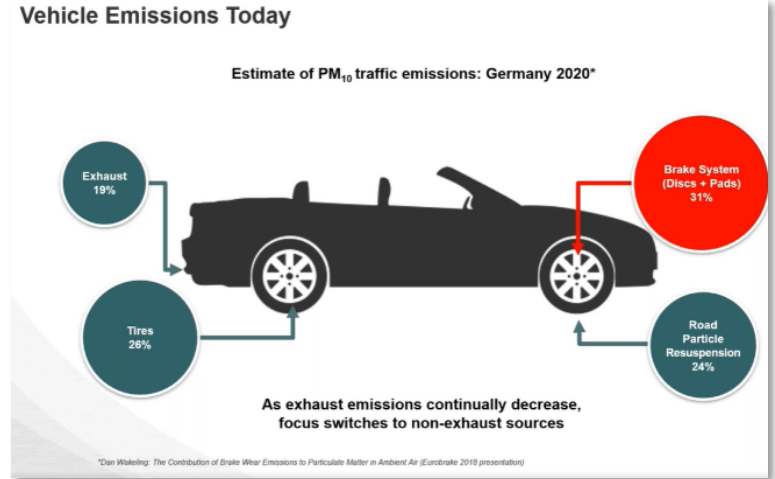
# Non-exhaust particulate emissions

Approx. **30% of  $PM_{10}$**  emissions are **caused by the brake system**.

BEV road wear and dust emissions are identical to ICEV with slightly more tire wear due to higher weight.

Much lower brakes wear by using recuperation braking but ...

**increased corrosion** problem **due to “underutilized” brake** components.



Photographs: Nitrex Metal Inc.

# MEASURES TO REDUCE BRAKE WEAR

Besides recuperation in EVs, specific software and filter systems, the brake system should be modified by **applying ceramic coating**

<https://www.bmb-consult.com/en/presscentre/2019/lowbrasys-the-clean-technology?msclkid=f911e8a3cf5f11ec9f608d6c4491e598>

- carbon-ceramics avoiding cast iron or steel discs (very expensive)
- coating of standard brake discs (moderate, less expensive)
- ferritic **nitrocarburizing of standard cast iron brake rotors** (most cost effective)

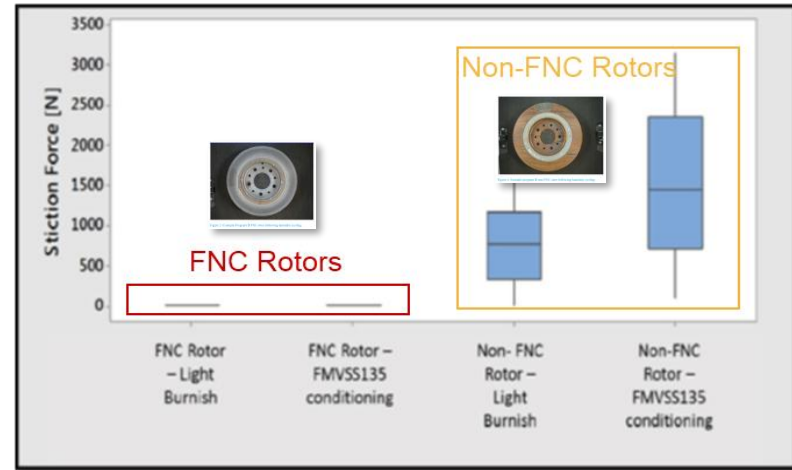


# Nitrocarburized rotors – a proven and reliable solution

*“By applying FNC GM was able to reduce brake-related warranty claims by over 70%”*

- High Performance and Durability*
- Enhanced Corrosion Resistance*
- Reduced Brake Dust*
- Improved Brake Lining (Pad) Wear*
- Increased Service Life from 40000 to 80000 miles*
- Reduced Pulsation*
- Reduced Pad Stiction***

<https://dev.safebraking.com/brake-tech-gm-fnc-rotors-the-cure-for-lot-rot-and-more>.



Robere, M., "Disc Brake Pad Corrosion Adhesion: Test-to-Field Issue Correlation, and Exploration of Friction Physical Properties Influence to Adhesion Break-Away Force," SAE Technical Paper 2016-01-1926, 2016, doi:10.4271/2016-01-1926.

Pad and rotor subjected to a 3 days of humidity cycling: 8 hours each day at 100% humidity at 38°C and 16 hours at 45% humidity at 25°C.

# FNC TECHNOLOGY

			
Housing and Sleeve for Offshore Drilling Equipment	Automotive Brake Adjuster	Rifle Cannon	Sludge Valve for Mining Industry

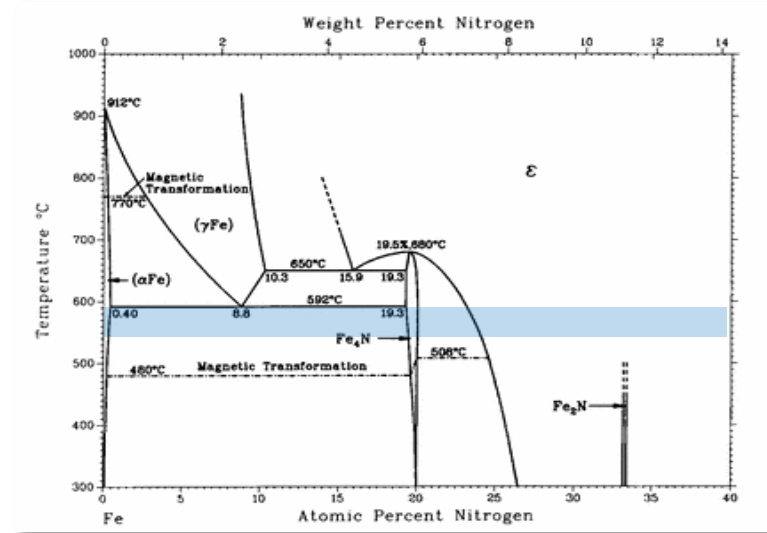
(Ferritic) Nitrocarburizing is mostly used on carbon steels and low alloy steels, but is also renowned for its benefit on hot work tool steels.

<https://www.nitrex.com/en/solutions/furnaces-technologies/nitriding-nitrocarburizing/nitreg-technologies/nitrocarburizing-nitreg-c/>

# Ferritic nitrocarburizing (FNC)

“Ferritic nitriding and nitrocarburizing are thermochemical case-hardening processes whereby **nitrogen (and carbon) are introduced** into the surface of a solid ferrous alloy, **in the ferritic state of the material.**”

Process temperature FNC: 560°C to 590°C

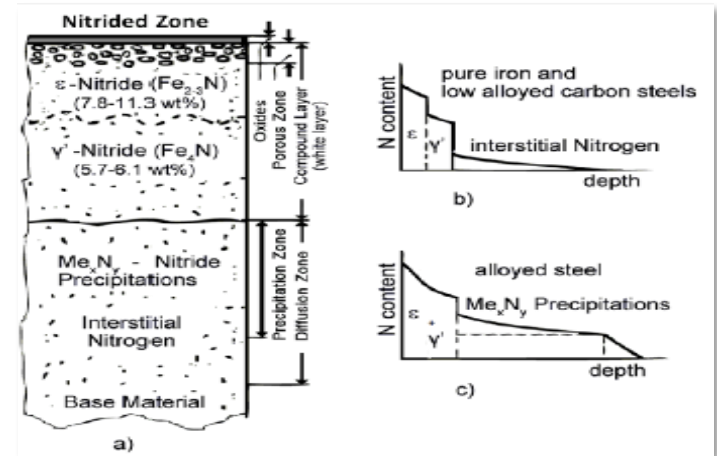


Acc. to H.A. Wriedt, N.A. Gocken and R.H. Nafziger, 1987

K. M. Winter, J. Kalucki, “Gas Nitriding and Gas Nitrocarburizing of Steel”, 2013, ASM Handbook, Volume 4A, Steel Heat Treating Fundamentals and Processes, Ed. Dossett and G.E. Totten, editors

# Ferritic nitrocarburizing (FNC)

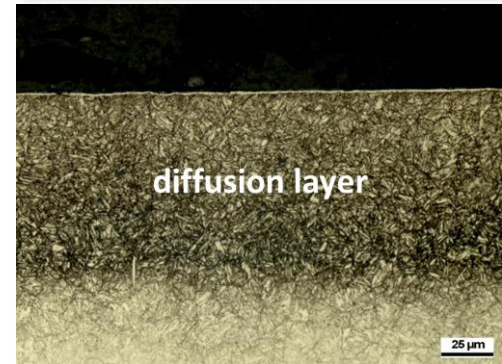
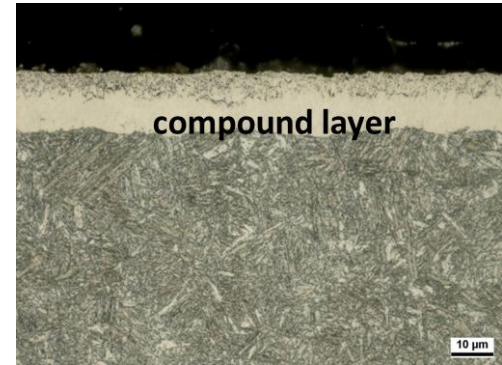
*“The nitrided layer created during treatment typically consists of a surface layer of iron nitrides and iron carbonitrides—referred to as a **compound layer** (also known as a white layer, due to its appearance in an etched micrograph). Underneath the white layer is a **diffusion zone**, which also contains carbonitrides of alloying elements.”*



K. M. Winter, J. Kalucki, “Gas Nitriding and Gas Nitrocarburizing of Steel”, 2013, ASM Handbook, Volume 4A, Steel Heat Treating Fundamentals and Processes, Ed. Dossett and G.E. Totten, editors

# Nitriding / Nitrocarburizing – different applications

- ❑ **Nitrocarburizing** is aiming for a **compound layer** of  $\text{Fe}_{2-3}\text{N}$  epsilon and  $\text{Fe}_4\text{N}$  gamma prime iron nitrides of high hardness, increasing corrosion resistance and lowering the coefficient of friction.
- ❑ **Nitriding** on the other hand is aiming for a comparably **deep hardened case** caused by Nitrogen diffusing into the iron lattice and forming nitrides with nitride forming alloying elements, such as Chromium, Titanium, Aluminum etc.

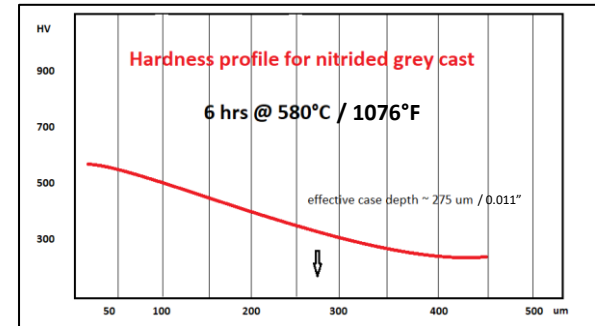
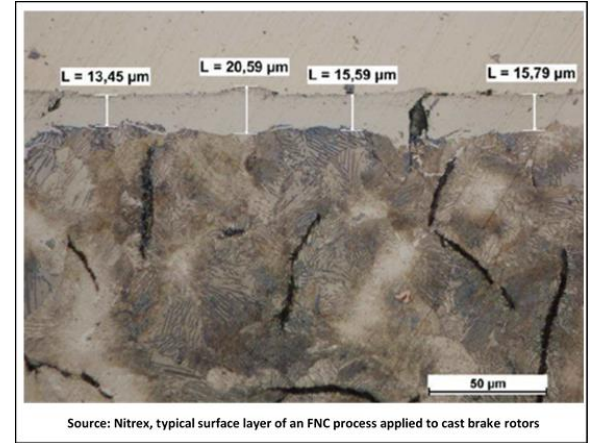


Courtesy to Leibniz-Institut für Werkstofforientierte Technologien  
IWT Bremen, Germany

# APPLYING FNC TO GCI ROTORS

FNC process is providing multiple advantages compared to untreated Grey Cast Iron rotors

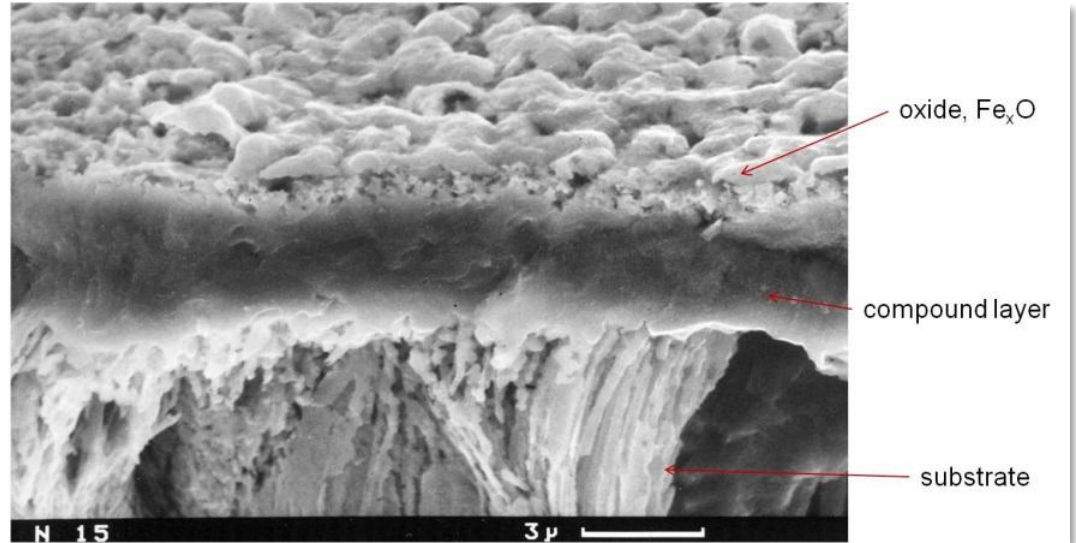
- ❑ Ceramic like **surface layer of** up to 30  $\mu\text{m}$  of **high hardness** ( $> 800 \text{ HV}_{0.05}$ ) (compound layer)
- ❑ Improved pitting resistance (diffusion zone)
- ❑ Improved fatigue life (diffusion zone)
- ❑ High **chemical resistivity** (compound layer)
- ❑ **Low coefficient of friction** (compound layer)
- ❑ **Improved heat resistivity** up to nitriding/nitrocarburizing temperature



# ONC – enhancing corrosion resistance properties

To enhance chemical resistance of the compound layer, nitrocarburized parts are often additionally oxidized.

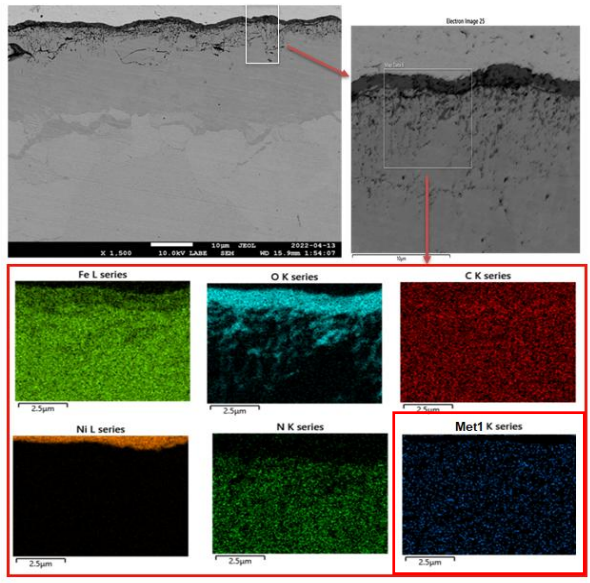
So-called **post-oxidation** applies a  $\text{Fe}_3\text{O}_4$  **Magnetite** layer on top of the compound layer.



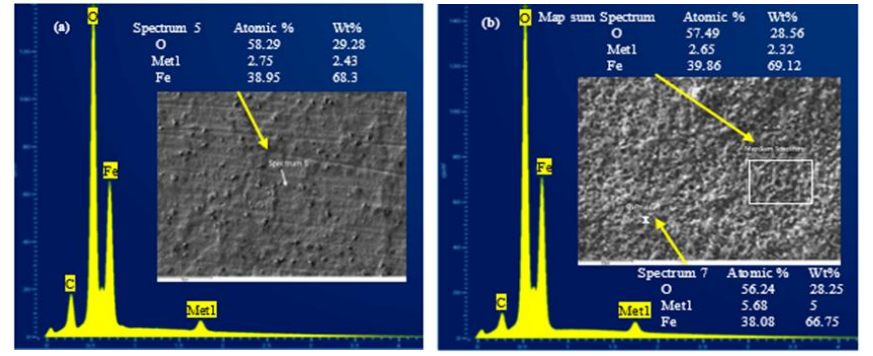
Courtesy to Leibniz-Institut für Werkstofforientierte Technologien - IWT Bremen, Germany

# Smart-ONC<sup>®</sup> – further improving corrosion resistance

SEM Cross – Section Maps



Chemical Composition of the coating on the surface by EDX



Surface morphology →

- ❑ Homogeneous magnetite phase layer
- ❑ Smart-ONC<sup>®</sup> chemical agent (2.7 At%).
- ❑ high concentration of Met1 indicates micropores are filled and sealed with amorphous Me1-Oxides.

❑ **Compound layer** with controlled porosity, **containing Me1**.

❑ A continuous, **compact oxide layer** on top of the nitrided layer, **containing Me1**.

# FNC of GCI rotors requires stress relieve (S/R) prior to nitrocarburizing

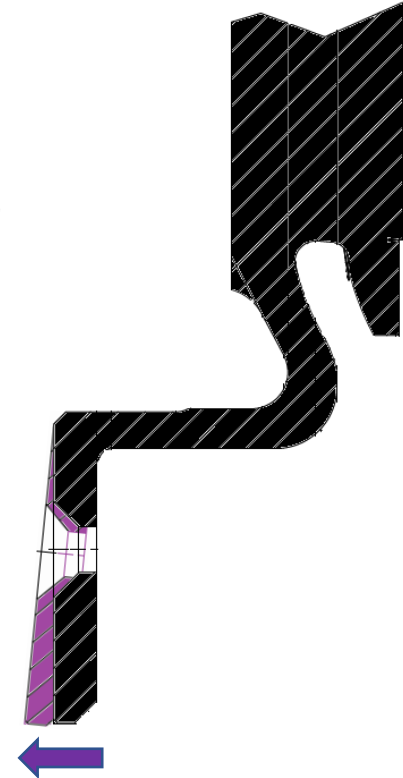
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**Casting stresses** are caused by uneven contraction of thinner and thicker parts during cooling.

When the rotors become hot, the stresses **are relieved** again, which leads to deformation causing lateral runout and deviations from concave/convex shape of the rotor head.

For this reason, the **rotors must be stress-relieved** at approx. 625°C before machining.

Advantage: after S/R and FNC, rotors are stable in shape and hardness (heat resistance) up to the FNC temperature.



# TESTING THE ROTORS



As cast, prior to S/R



after S/R



as machined



after FNC



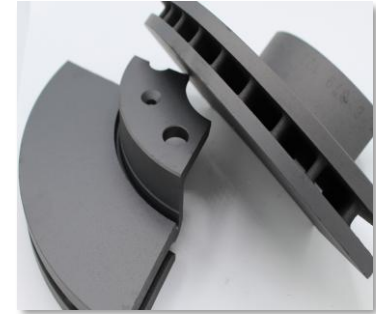
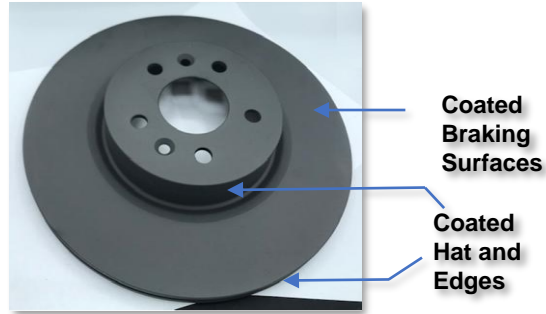
FNC/ONC



FNC/Smart-ONC

Standard GCI rotors applying S/R at 630 °C and FNC at 570 °C

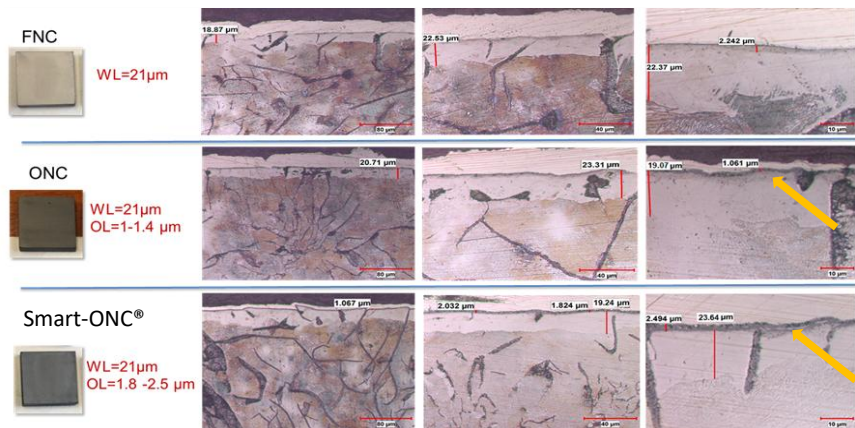
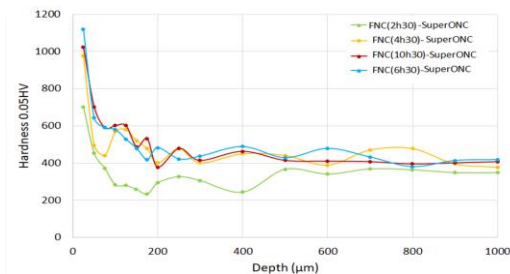
# Appearance after FNC, FNC+ONC and FNC plus Smart-ONC®



- The rotor is **fully coated inside and outside**.
- Inner vanes and all exposed surfaces.

# Metallography, hardness, roughness

Part name/ steel grade	Treatment	Treatment Name	White layer Thickness ( $\mu\text{m}$ )	Oxide layer Thickness ( $\mu\text{m}$ )	Porosity (%)	Hardness (HV) (50g load)	Hardness (HV) (100g load)	Hardness (HV) (1kg load)
Brake discs/cast iron	Untreated	GCI	-	-	-	605	519	242
	FNC	FNC	19-23	-	$\leq 15$	815	798	546
	FNC-ONC	ONC	19-23	1-1.4	$\leq 15$	741	725	514
	FNC-Smart ONC	Smart ONC	19-23	1.5 - 2.5	$\leq 15$	745	732	529



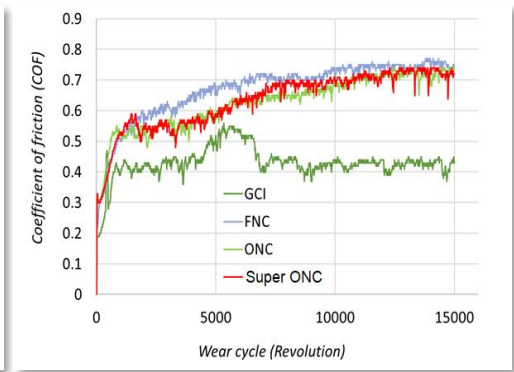
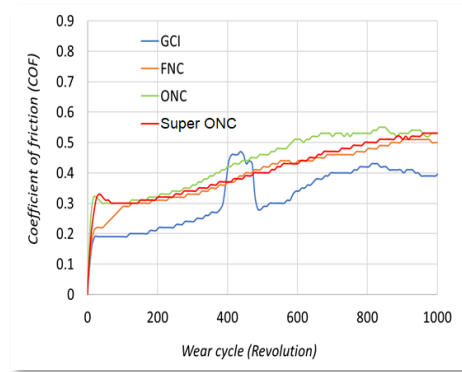
Roughness				
Treatment name	Ra	Rz	Rq	Rt
GCI	0.868	4.792	1.018	6.755
FNC	1.152	7.294	1.571	9.043
ONC	1.255	7.018	1.569	9.356
Smart-ONC®	1.289	6.927	1.524	9.032

Roughness increase might be related to graphite flakes sticking out of the compound layer; it will be quickly removed by first braking.

# Pin-on-disc – COF coefficient of friction

## Pin-on-disc wear testing

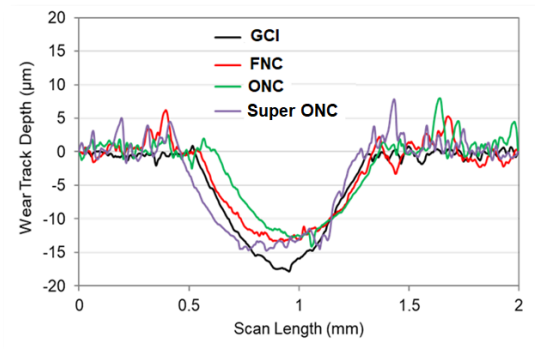
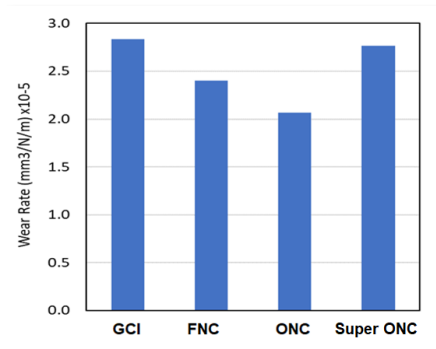
- ❑ load 20 N and 5 N
- ❑ Rotational speed 500 rpm
- ❑ Linear speed of 250 mm/s
- ❑ Wear track diameter of 9.5 mm.
- ❑ 15 thousand cycles
- ❑ wear distance 448 m
  
- ❑ The counter face: 6 mm Alumina ball
- ❑ Contact stress (calc.) 1.37 GPa



# Pin-on-disc – micro wear rate

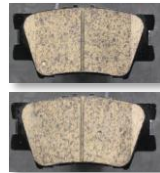
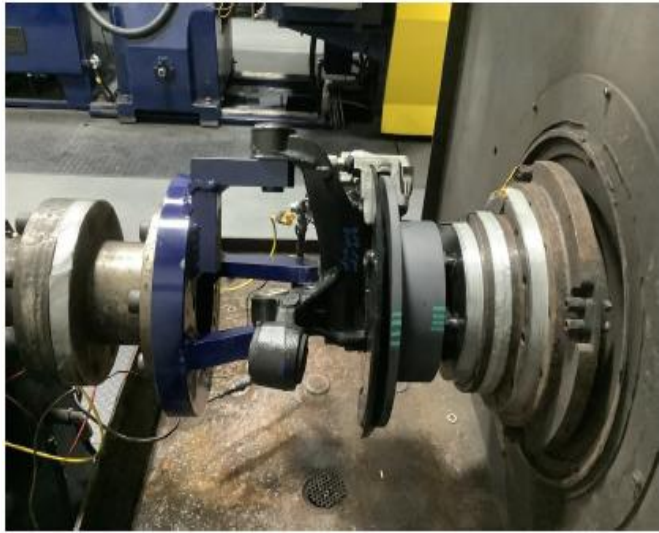
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Sample	Worn Area	Wear track perimeter	Wear volume mm <sup>3</sup>	Wear Rate 10 <sup>-5</sup> mm <sup>3</sup> /N/m
GCI	0.0085	29.85	0.25	2.83
FNC	0.0072	29.85	0.21	2.40
ONC	0.0062	29.85	0.19	2.07
Smart-ONC®	0.0083	29.85	0.25	2.77

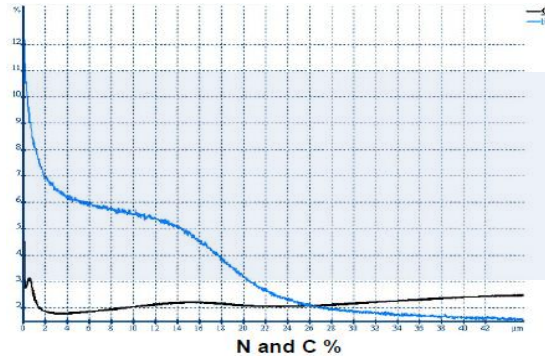
# Dynamometer – SAE J2707B, block wear test including high-speed (180 km/h)



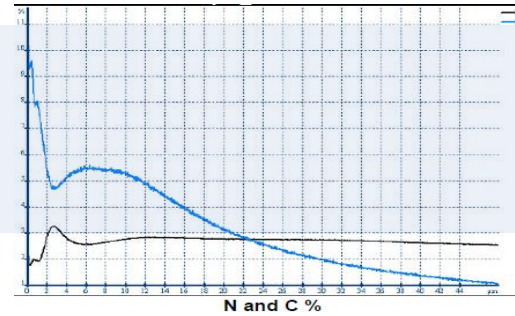
Smart-ONC® coated discs combined with NAO pads

Cycle No	Cycle test	Initial speed Km/h	Release speed km/h / deceleration m/s <sup>2</sup>	Initial Brake temperature
(I)	Burnish	50	4.03 / 2.59	100
(II)	Town block #1	50	4.03 / 2.59	150
(III)	Highway block #1	150	80 / 4.12	150
(IV)	Country road block #1	80	4.03 / 3.56	200
(V)	Country road block #2	100	4.03 / 4.06	125
(VI)	<b>Highway block #2</b>	<b>180</b>	80 / 5.11	100
(VII)	Town block #2	50	4.03 / 2.59	150
(VIII)	Country road block #3	100	4.03 / 4.06	125
(IX)	Hill descent block	80	4.03 / 3.57	350

# Dynamometer – SAE J2707B, block wear test including high-speed (180 km/h)



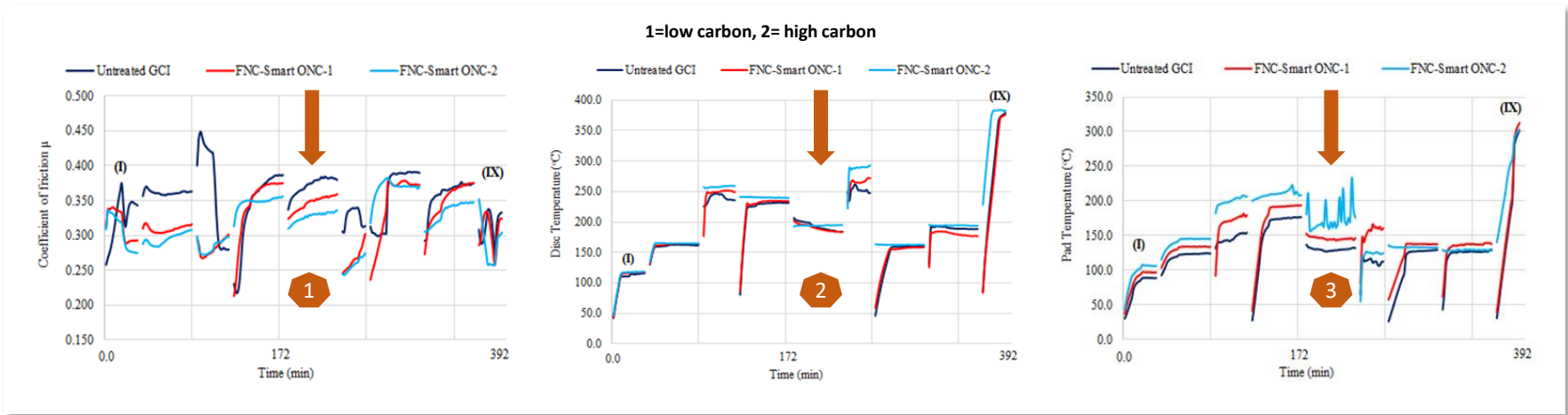
No decarburization of the nitrided layer



We treated **two different versions** to compare temperature stability and corrosion behavior of the compound layer

- Low carbon (left)
- High carbon (right)

# Dynamometer – SAE J2707B, block wear test including high-speed (180 km/h)



- ❑ Stopping distance was not affected by FNC / Smart-ONC<sup>®</sup> coating
- ❑ While COF (1) of Smart-ONC<sup>®</sup> variants start lower they match GCI after Highway block 2
- ❑ 2) No significant difference in disc temperatures (2) indicating no change of thermal properties by applying FNC
- ❑ 3) Higher pad temperatures (3) with Smart-ONC<sup>®</sup> variants until end of Highway block 2

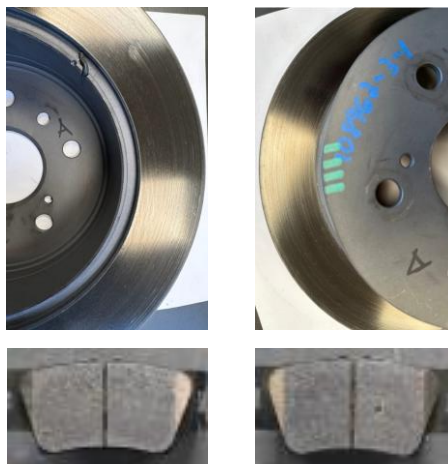
Assumption: creating **friction surface changes** by pad material pickup

# Dynamometer – SAE J2707B, appearance after testing

Untreated GCI



FNC/Smart-ONC® Low Carbon



FNC/Smart-ONC® High Carbon

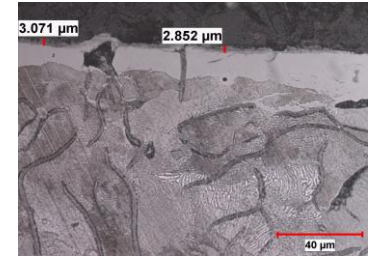
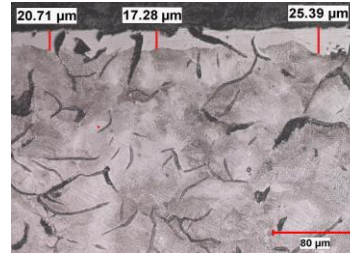


- Grooves on the untreated GCI disc
- No wear on the coated discs
- Low wear on brake pads
- No visible cracks on the disc surface
- Pad material accumulation** on FNC/Smart ONC® discs, more pronounced on the high carbon variant

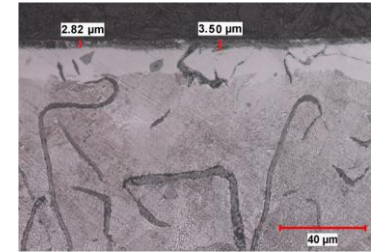
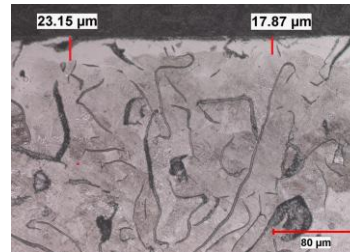
Brake disc surface finishing	Rotor (g)	Outboard Pad (g)	Inboard Pad (g)
Untreated CGI	0.1	2.8	2.5
FNC/Smart ONC® LC	0	4.1	4.7
FNC/Smart ONC® HC	0	4.5	7.3

# Dynamometer – SAE J2707B, micrographs after testing

FNC-Smart ONC®-Low Carbon



FNC-Smart ONC®-High Carbon

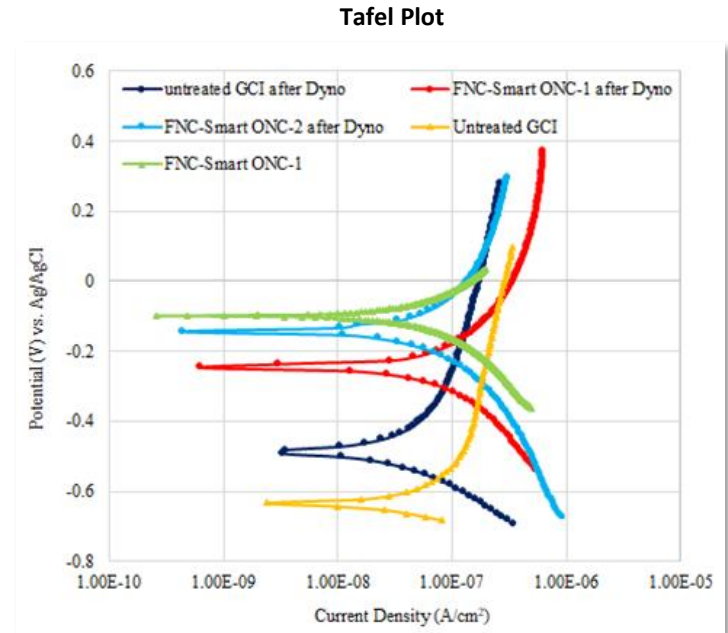


- ❑ No delamination and **no cracks** appeared **in the compound layer** of the brake disc
- ❑ Appearance of a thin **transfer film from the friction material** on top of the compound layer

## Corrosion behavior – before and after block wear testing

Electrochemical corrosion measurement prior and after after block wear testing:

- ❑ **untreated GCI** showed worst corrosion behavior, slightly **improving after testing**, most likely by picking up pad material.
- ❑ **Smart-ONC® LC version** shows highest corrosion resistance but **looses after testing** due to material losses of oxide and compound layer. **HC variant** stays almost constant.



Potentiostat - Galvanostat UriScan model PG581  
Saturated calomel electrode (SCE) Ag/AgCl reference  
5% NaCl (pH = 6.4; T= 23°C)

# Cyclic corrosion – SAE J2334, after 36 cycles (2.25 years)

- ❑ Smart-ONC® versions (right) of **solid discs** display less corrosion on the friction surface compared to traditionally post-oxidized version (left)

- ❑ **Ventilated discs** start corroding on the non-machined surface, preferably on sharp edges and in the ventilation



- ❑ Untreated rotors will be fully corroded

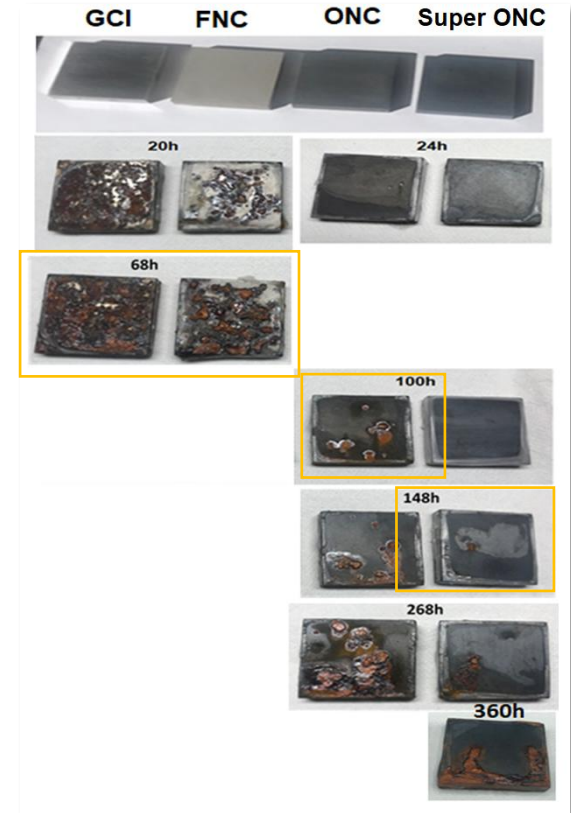


FNC-LC / ONC

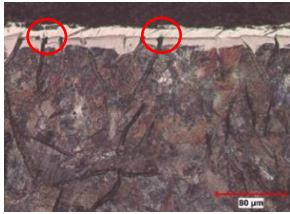
FNC-LC / Smart-ONC®

# Salt spray corrosion – ASTM B 117, samples from friction surface

- ❑ The GCI rotor is totally corroded after 20 hours.
- ❑ Nitrocarburized rotor survives 48 to 68 hours
- ❑ Post-oxidized variant quadruples this time
- ❑ **Smart-ONC®** is at least two times as effective as standard ONC and at least **eight times as effective as FNC** variants.



# Salt spray corrosion – ASTM B 117, $\frac{1}{8}$ of a rotor cut from ventilated discs



Compound layer defects caused by graphite flakes (red circles) and remnants of the casting mold

24 h



120 h

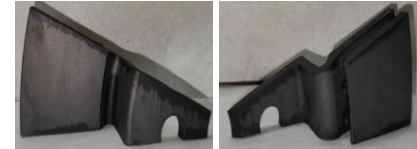


168 h

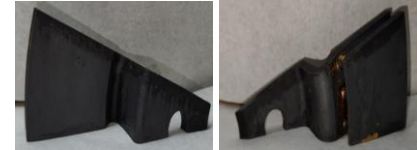


Addressing as cast, non-machined surfaces

48 h



170 h



216 h



340 h



# Comparing FNC / Smart-ONC<sup>®</sup> to painted and non-thermal coated versions

## Nitrex Smart-ONC<sup>®</sup> vs.

- Untreated GCI
- Painted GCI
- Delphi Technologies coating  
*zinc-flake-based electroless coating*



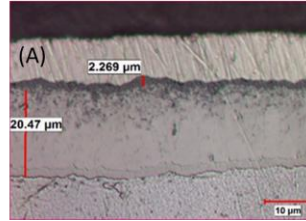
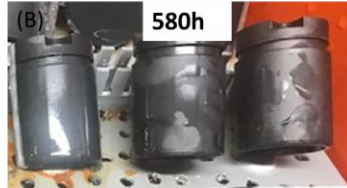
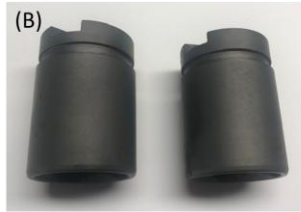
<https://www.vehicledynamicsinternational.com/news/braking/delphi-reports-strong-corrosion-resistance-in-brake-discs.html>

# NITREX FNC + SMART-ONC® SUMMARY

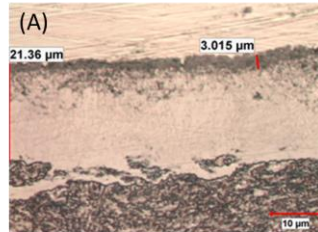
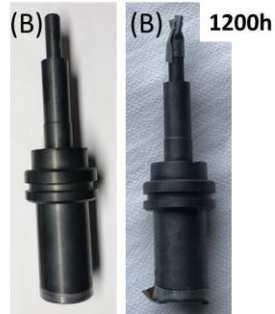
The findings indicate promising outcomes for Smart-ONC® treated rotors.

- ❑ FNC, FNC / ONC as well as FNC / Smart-ONC does not impact braking performance.
- ❑ Untreated and FNC rotors show only low corrosion resistance.
- ❑ ONC post-treatment shows an increase but does not entirely prevent the appearance of corrosion spots within 24 hours of exposure.
- ❑ Smart-ONC® treatment demonstrates notably improved corrosion resistance. The initial appearance of rust spots occurs after 170 hours of salt spray exposure.
- ❑ Smart-ONC® effectively halts the spread of corrosion, likely owing to its self-recovery property.

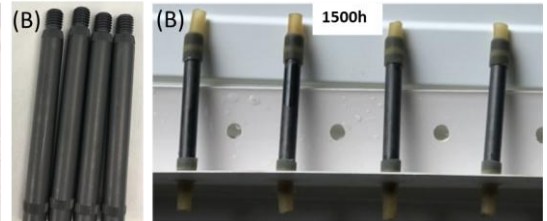
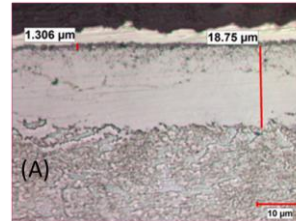
# Applying Smart-ONC® to other applications



Brake pistons  
AISI 1045

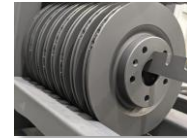
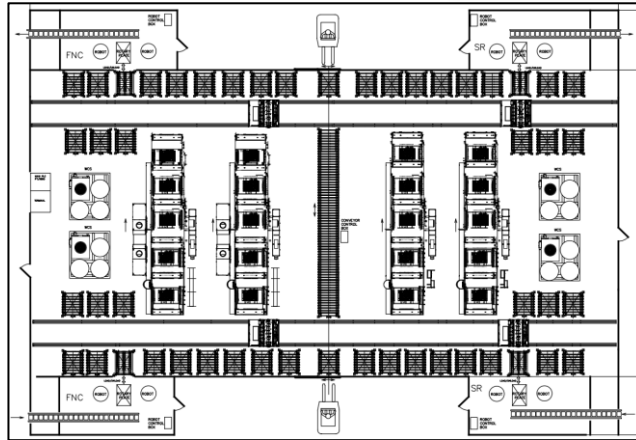
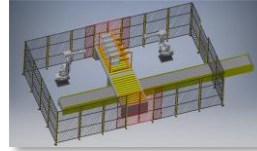
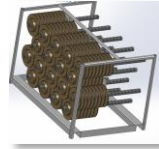
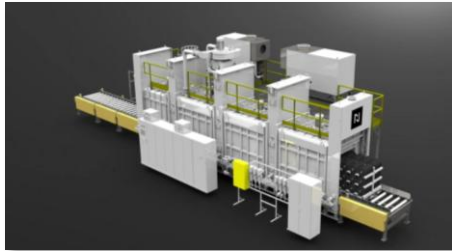


Carbon steel part with sharp edges



Wiper shafts – AISI 1045

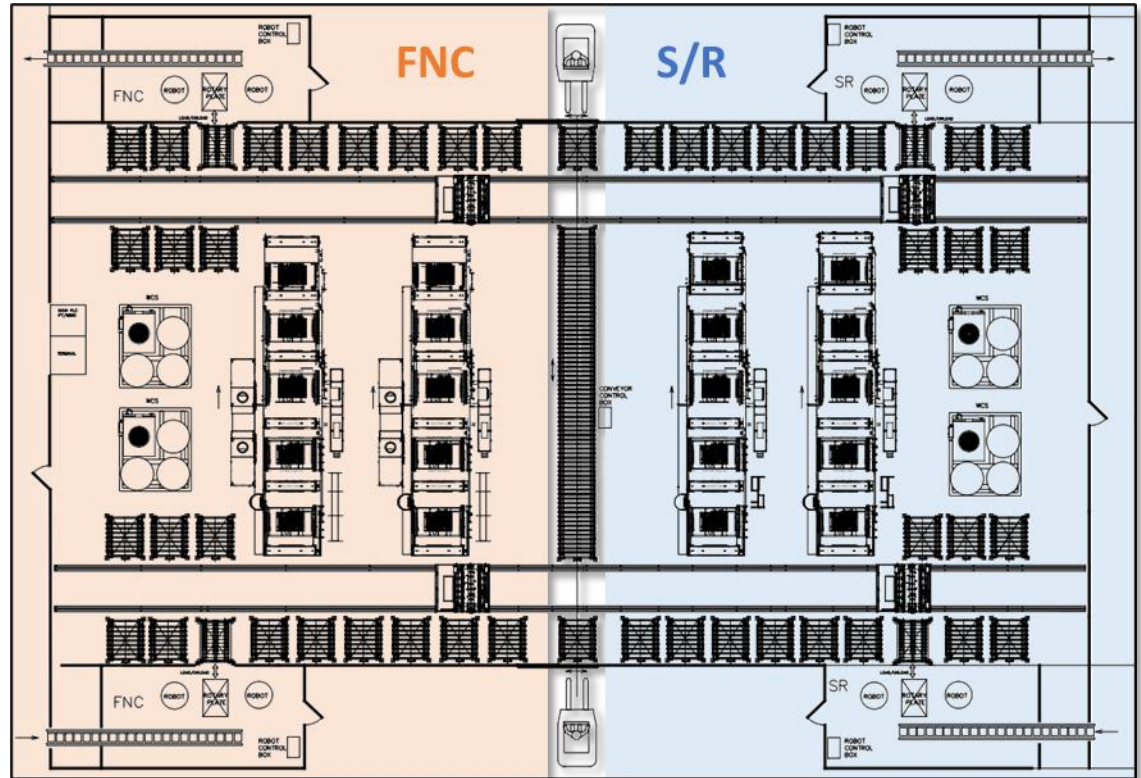
# INDUSTRIALIZATION



# Highly redundant automated “lights-out” heat treating cell

## Taking on mass-production

- ❑ 2 multi-chamber S/R furnaces
- ❑ 2 multi-chamber FNC furnaces
- ❑ 8 robots
- ❑ 4 charge cars on 2 tracks
- ❑ Transverse line



# Multi-chamber S/R & FNC furnaces

## Matching performance and stroke-time

- 4 or 5 sealed chambers
  - Heating
  - 1<sup>st</sup> FNC / S/R stage
  - 2<sup>nd</sup> FNC / S/R stage
  - Smart-ONC<sup>®</sup> stage / 3<sup>rd</sup> S/R stage
  - Fast cooling

Auxiliaries such as closed loop cooling,  
exhaust neutralizer, gas supply ..

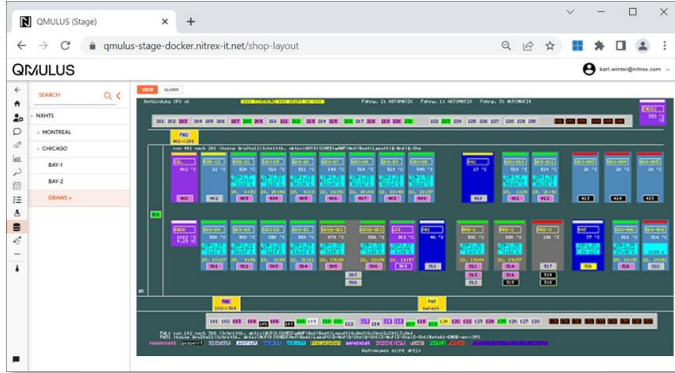


Nitrex multi-chamber furnace for high volume FNC with ONC option.

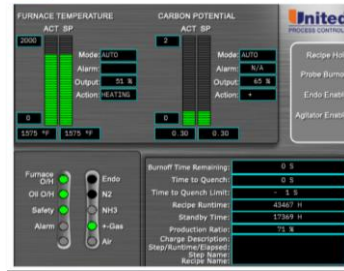
# PERFORMANCE

Maximum load weight:	3 t
Operating time:	24/7
Operating days:	350, allowing for 15 days without production
Availability:	90 %
Average weight / rotor:	12 kg as cast, 9 kg machined
Typical performance / year:	1.5M fully processed rotors S/R & FNC

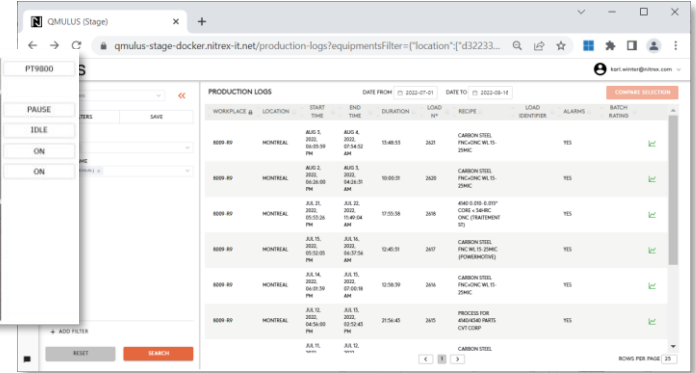
# SUPERVISION & CONTROLS



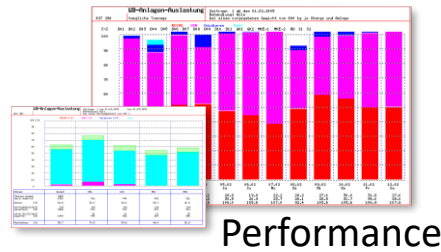
Cell view



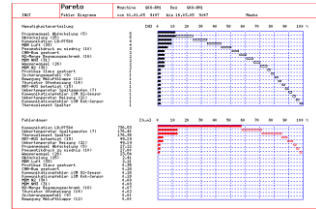
Furnace view



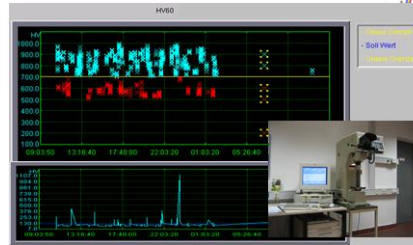
Production logs



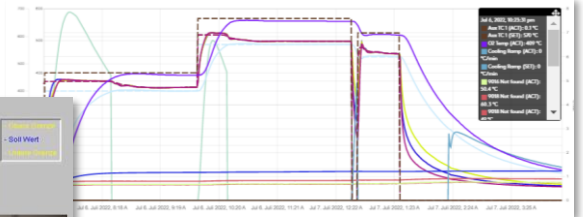
Performance



Alarm statistics



QA lab results



Process chart of a load

## Contact Info

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Thank you for your attention!

Marcin Stoklosa  
NITREX METAL sp. z o.o  
Jedności 48, 41-218 Sosnowiec  
+48 509 93 1 370 (mobile)  
marcin.stoklosa@nitrex.com

