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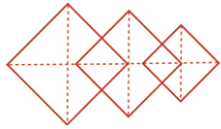
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Sébastien JÉGOU, Benjamin GUILLOT, Laurent BARRALLIER - Activation of surfaces prior to gaseous nitriding of a 3wt.% Cr carbon iron-based alloy - In: Nitriding Symposium 4, Etats-Unis, 2016-11-17 - Proceedings of the 4th Nitriding Symposium - 2016

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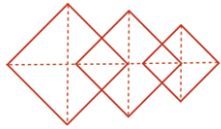




Activation of surfaces prior to gaseous nitriding of a 3wt.% Cr carbon iron-based alloy

Speaker: Sébastien Jégou, Associate Professor
Arts et Métiers ParisTech, MSMP Laboratory
France

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B.Guillot, PhD, Arts et Métiers ParisTech, MSMP Laboratory



2009 – PhD – Arts et Métiers ParisTech, Aubert & Duval, France

- Gaseous Nitriding
- Residual Stresses



2010 – PostDoc – DTU, Denmark

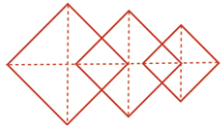
- Expanded Austenite
- Nitriding / Carburizing
- Residual Stresses



2011-... - Associate Professor – Arts et Métiers ParisTech, France

- Materials Science
- Surface Engineering

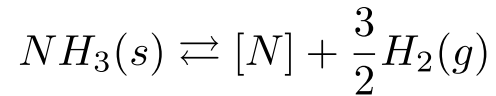




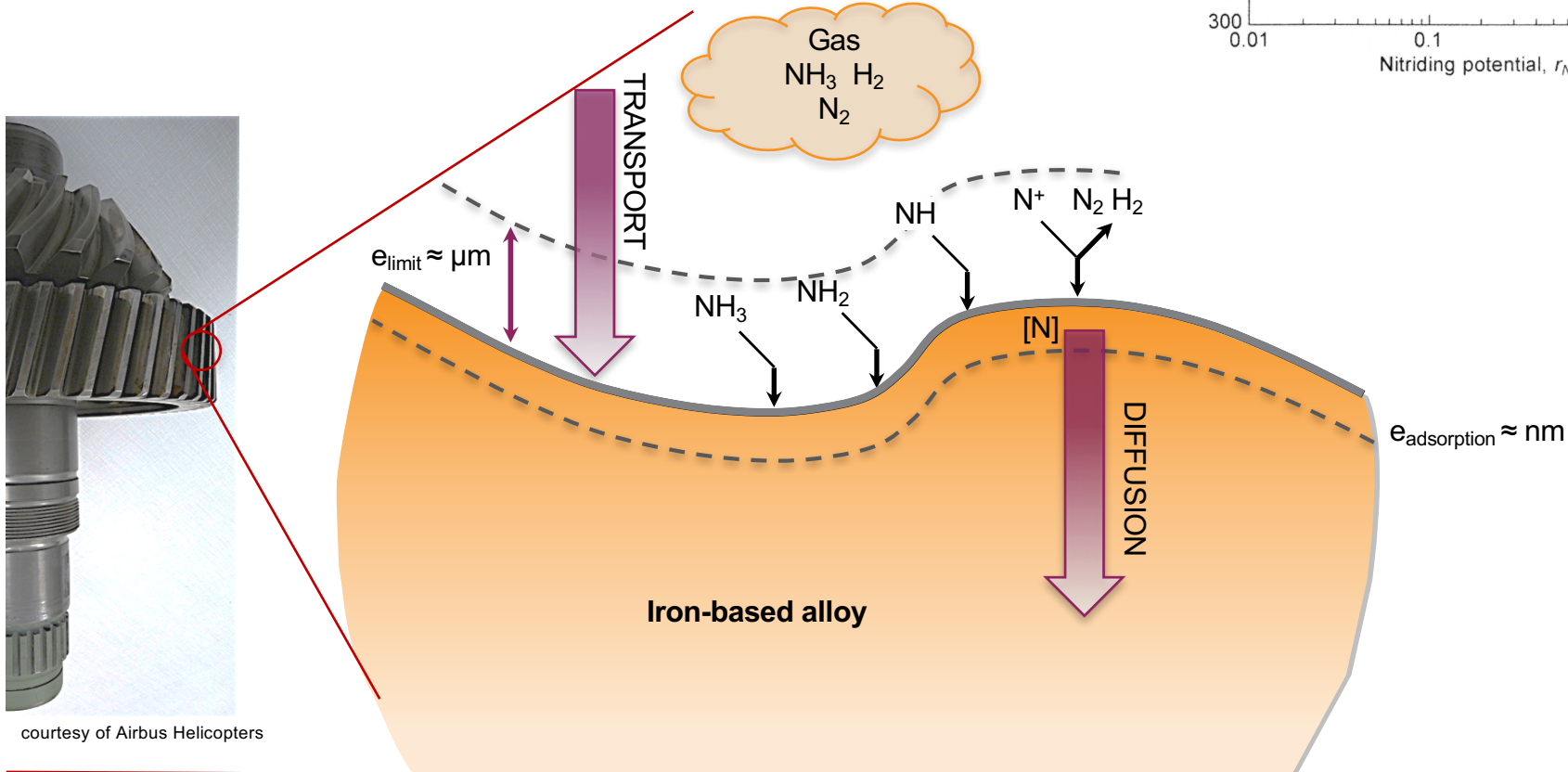
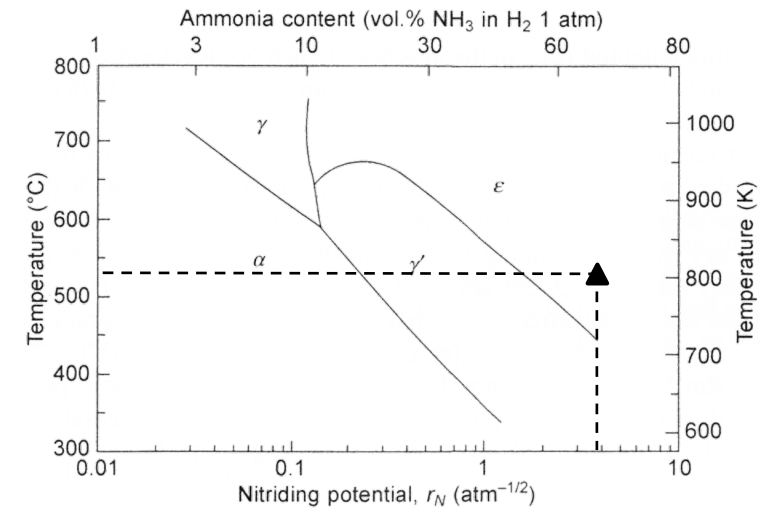
AGENDA

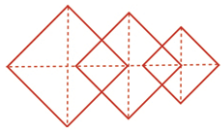
1. Introduction
2. Experiments
3. Results
4. Conclusion

1. Introduction - Context

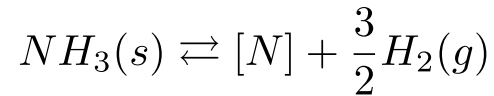


$$K_N = \frac{p_{NH_3}}{p_{H_2}^{3/2}}$$

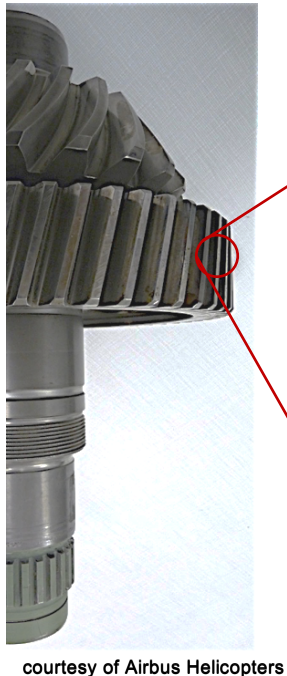
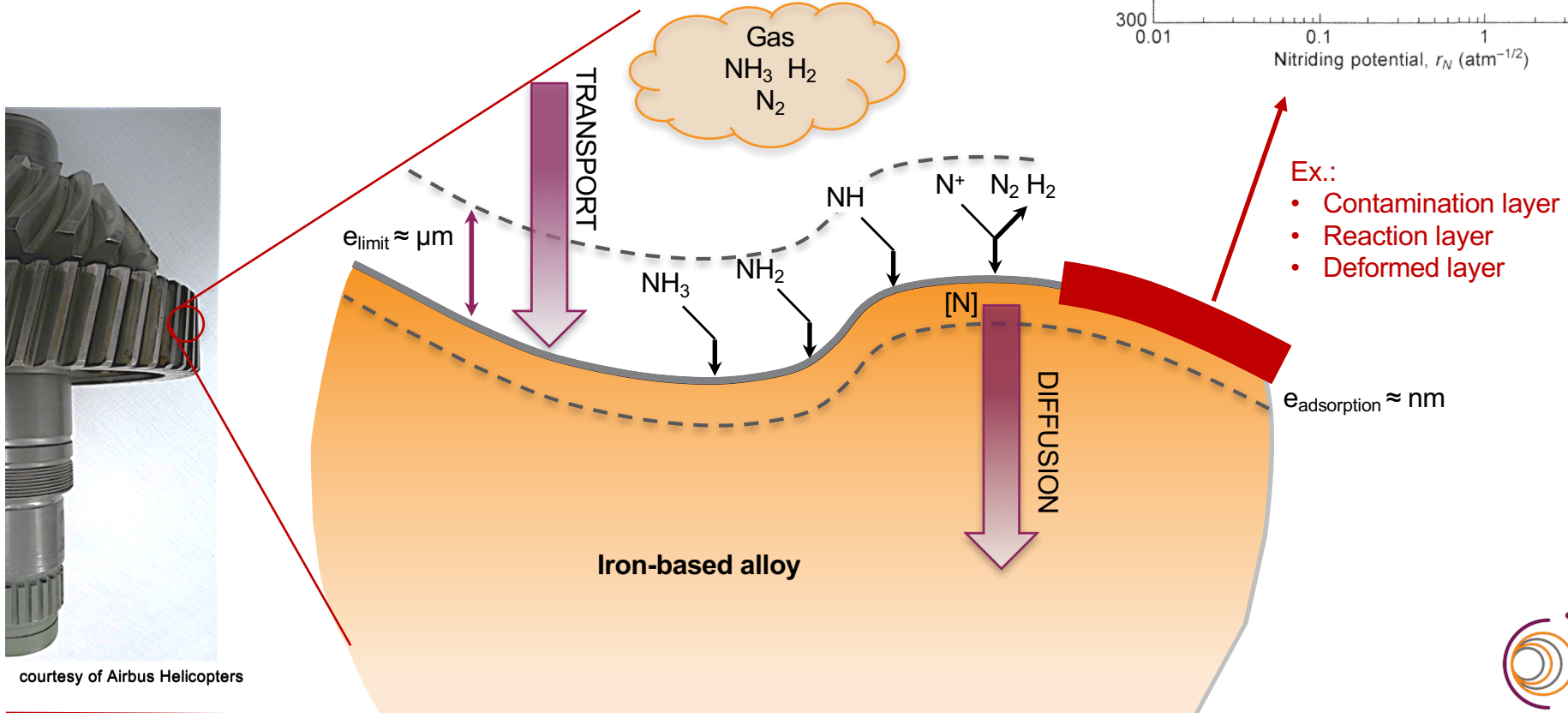
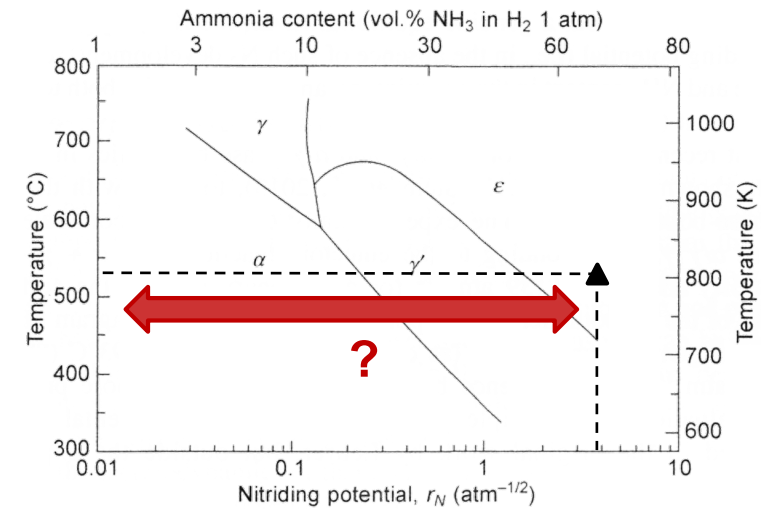


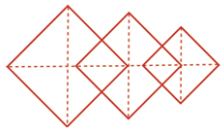


1. Introduction - Context



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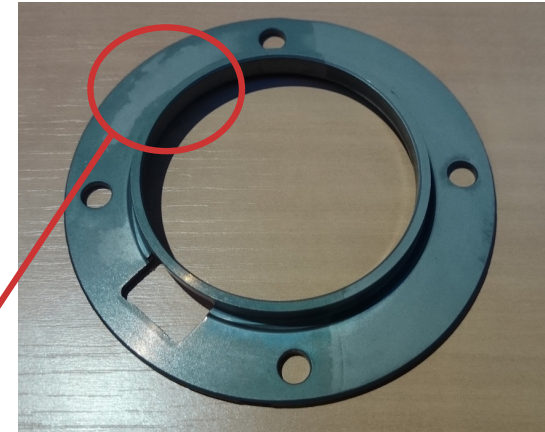




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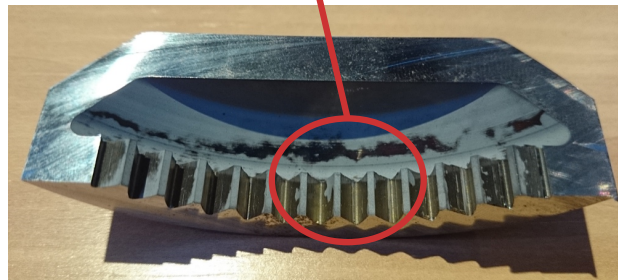


courtesy of Airbus Helicopters



courtesy of Bodycote

- Soft spot
- Lack of nitriding
- Heterogeneous nitriding

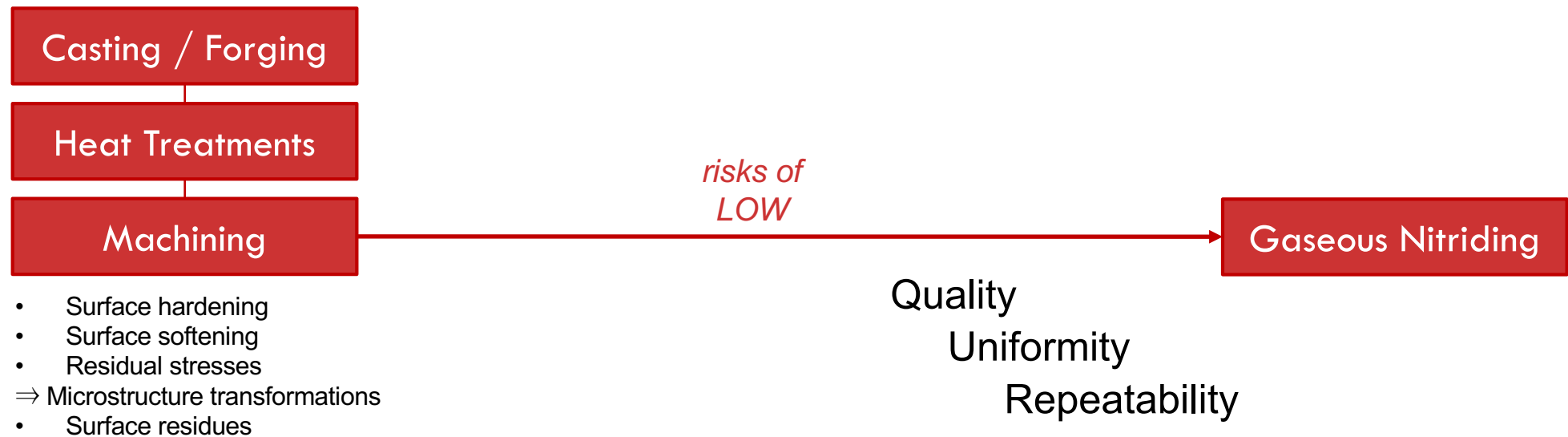


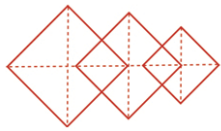
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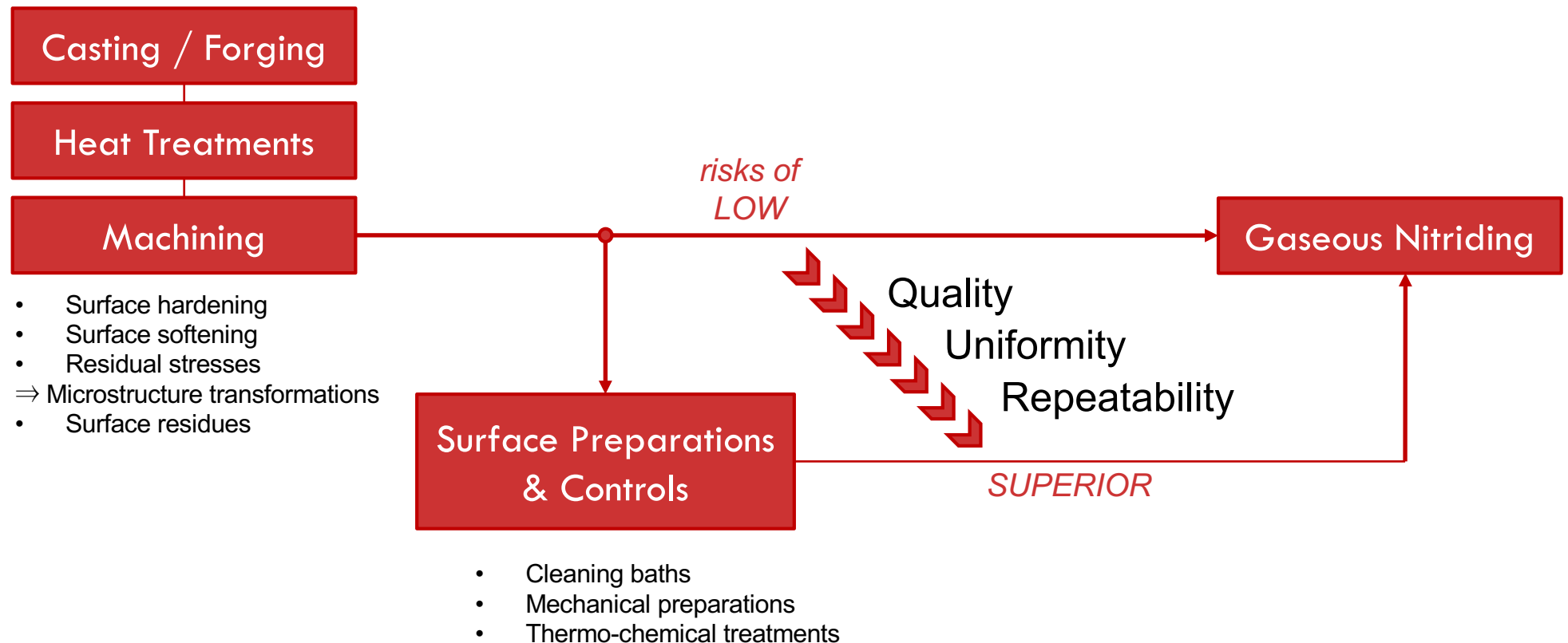


1. Introduction - Context





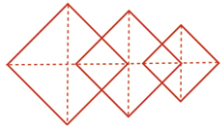
1. Introduction - Context



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Surface Preparations & Controls

- Cleaning baths: residues from
 - Machining oil (sulphate, phosphate, silicon, ...)
 - Cleaning baths (anionic surfactant (sulphonates, sulphates...))



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 - Machining oil (sulphate, phosphate, silicon, ...)
 - Cleaning baths (anionic surfactant (sulphonates, sulphates...))
- Mechanical preparations: Sandblasting (sanding)
 - Passivation/corrosion layer
 - Heterogeneous metallurgy/microstructure

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 - Passivation/corrosion layer
 - Heterogeneous metallurgy/microstructure
- Thermo-chemical treatments: **controlled** layer (nature, thickness)
 - Oxidization
 - Phosphating (Zn, Mn)
 - ...

⇒ Surface homogeneity visual control

⇒ Protection from (heterogeneous) surface reactions

⇒ Activation of the NH_3 decomposition

1. Introduction - Context

Surface Preparations & Controls

- Thermo-chemical treatments: **controlled** layer (nature, thickness)
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 - ⇒ **Activation** of the NH_3 decomposition
 - Oxidization (NH_3 decomposition catalyst)
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Surface Preparations & Controls

- Thermo-chemical treatments: **controlled** layer (nature, thickness)
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 - ⇒ **Protection** from (heterogeneous) surface reactions
 - ⇒ **Activation** of the NH_3 decomposition
 - Oxidization (NH_3 decomposition catalyst)
 - Phosphating (Zn, Mn)
- ⇒ Better Process Flexibility: **In-situ pre-treatments**
 - cleaning
 - activation
- Oxidization : oxygen reactivity with contaminants
- Urea

$$\text{NH}_2 - \text{CO} - \text{NH}_2(aq, s) \rightarrow \text{HNC}(\text{O})(g) + \text{NH}_3(g) \rightarrow \text{CO}_2(g) + \text{NH}_3(g)$$
- NH_4Cl

$$\text{NH}_4\text{Cl}(aq, s) \rightarrow \text{HCl}(g) + \text{NH}_3(g)$$

⇒ Heating stage: atmosphere of Acids + N-adsorption

2. Experiments

- **Material: 33CrMoV12-9**
 - 17 x 13 x 5 mm³
 - Austenitized @ 920 °C, 90 min
 - Oil quenched
 - Tempered @ 640 °C, 1 h

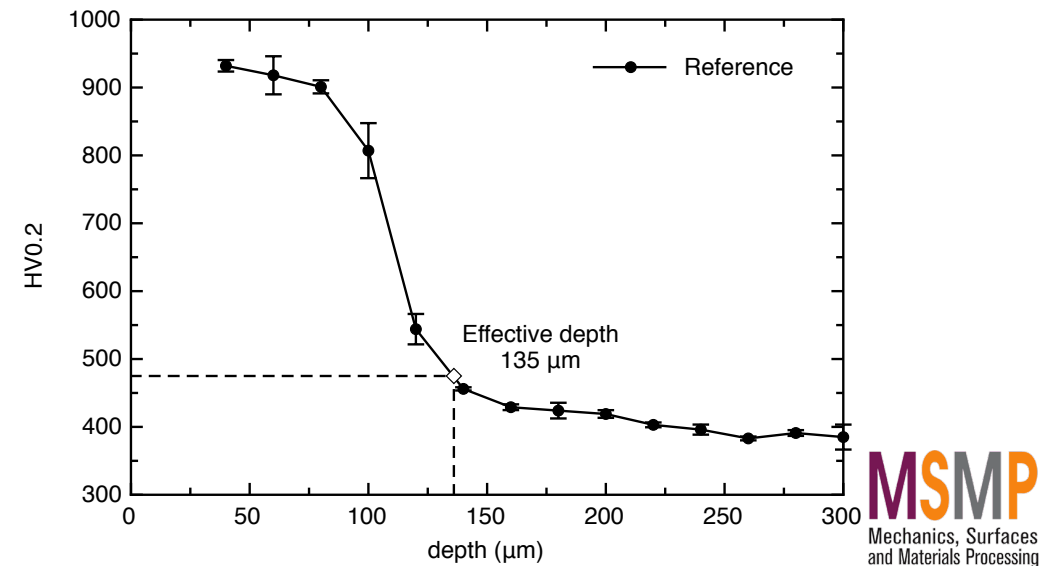
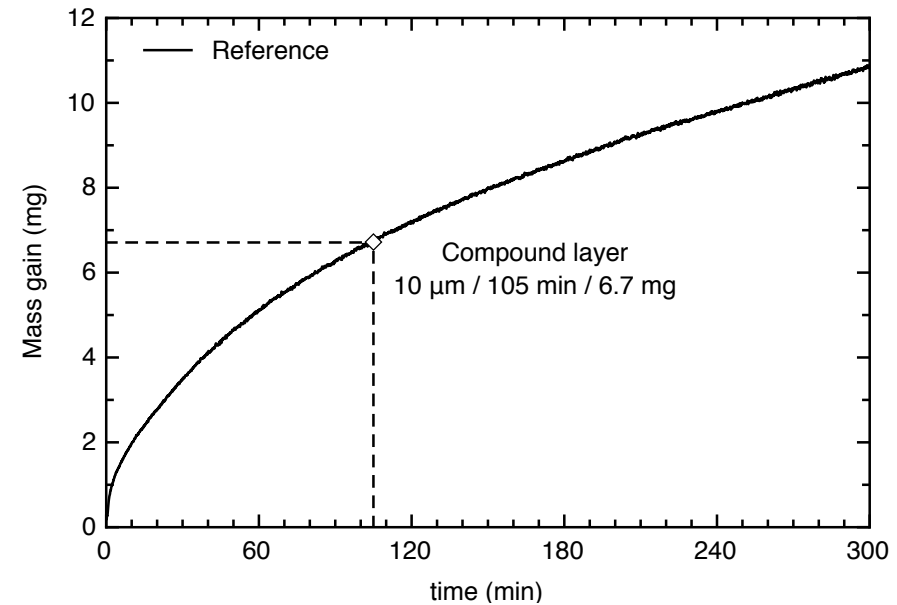
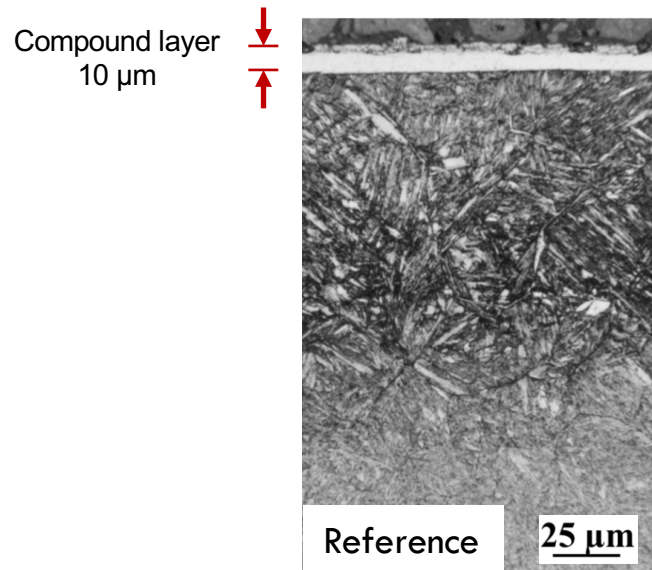
Composition (wt.%)					
C	Cr	Mo	V	Mn	Fe
0.32	2.97	0.84	0.28	0.55	bal.

- Sample preparation:
 - Degreased
 - Rinsed in water
 - Dried in alcohol

- **Surface contamination:**
 - **Water-dissolved machining oil**
 - 1 to 100 vol.%
 - 1 min dipping
 - Droplets removed
- **In-situ treatments:** 350-400 °C, 1 h
 - Vacuum Stages
 - Oxidization (O₂)
 - Urea / NH₄Cl
 - 200 mg
 - Neutral atmosphere
- **Gaseous Nitriding:**
 - Thermogravimetric analyser (Setsys Evo.)
 - Vacuum Stages
 - Heating/Cooling @ 10 °C.min⁻¹ under N₂
 - **520 °C, 5 h, K_N 3.7 atm^{-1/2}**
 - NH₃-N₂-H₂ (200 mL.min⁻¹)

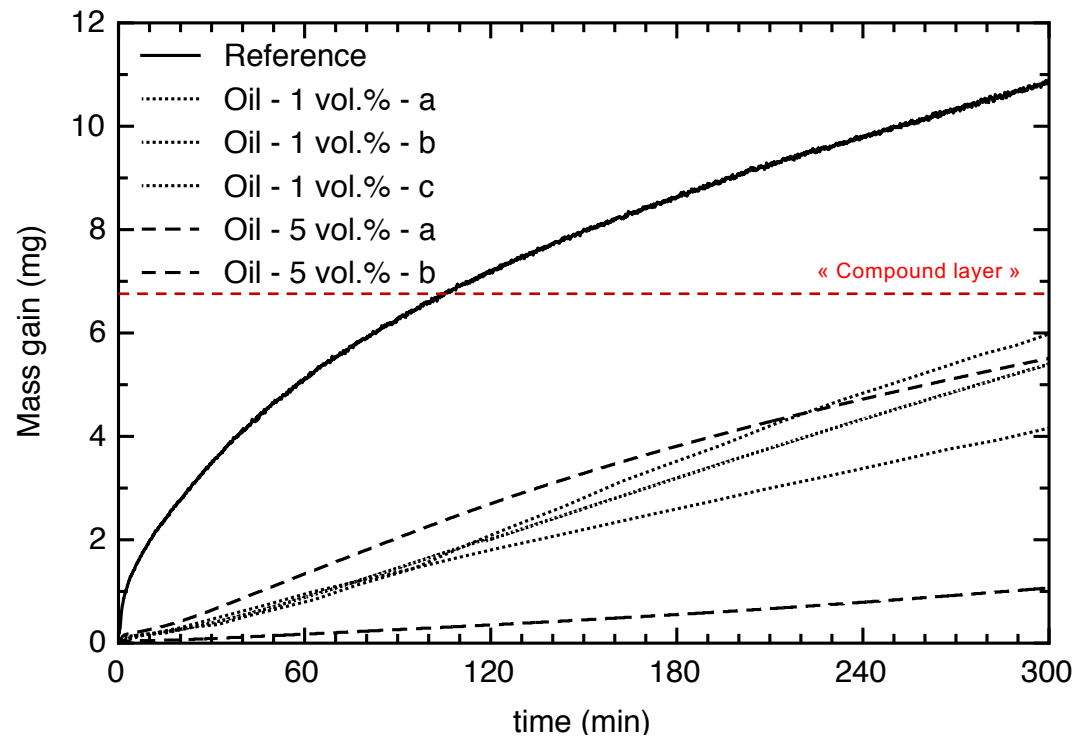
3. Results

0. The reference



3. Results

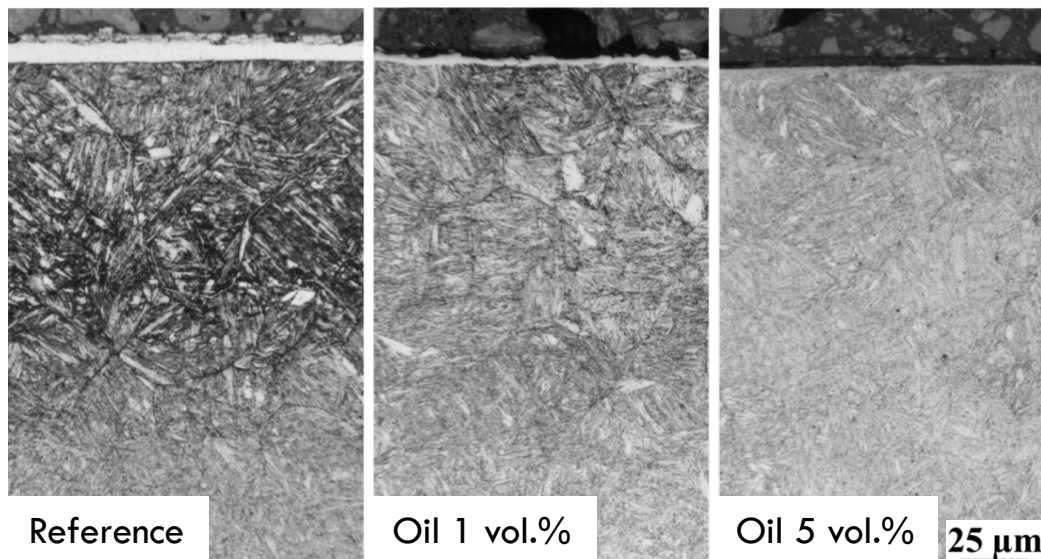
a. Influence of water-dissolved oil contaminations



Sample	Mass gain after nitriding (mg)
Reference	10.89 ± 0.33
Oil 1 vol.% - a	5.39 (49.5 %)
Oil 1 vol.% - b	5.98 (54.9 %)
Oil 1 vol.% - c	4.16 (38.2 %)
Oil 5 vol.% - a	1.07 (9.8 %)
Oil 5 vol.% - b	5.51 (50.6 %)

3. Results

a. Influence of water-dissolved oil contaminations

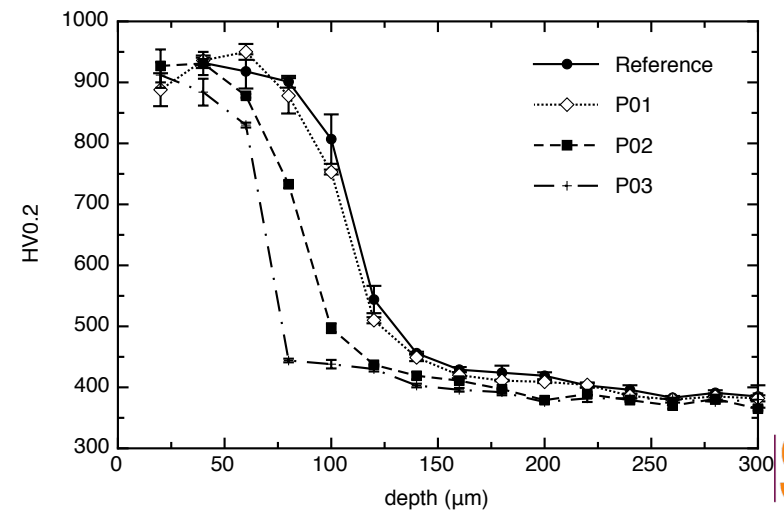
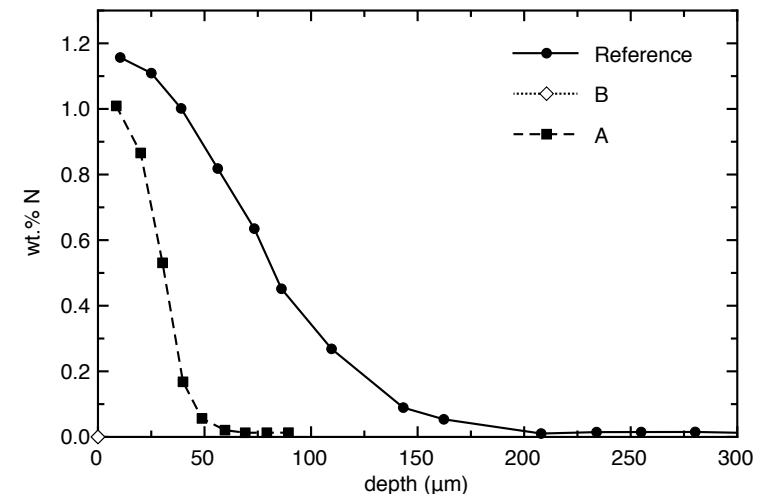
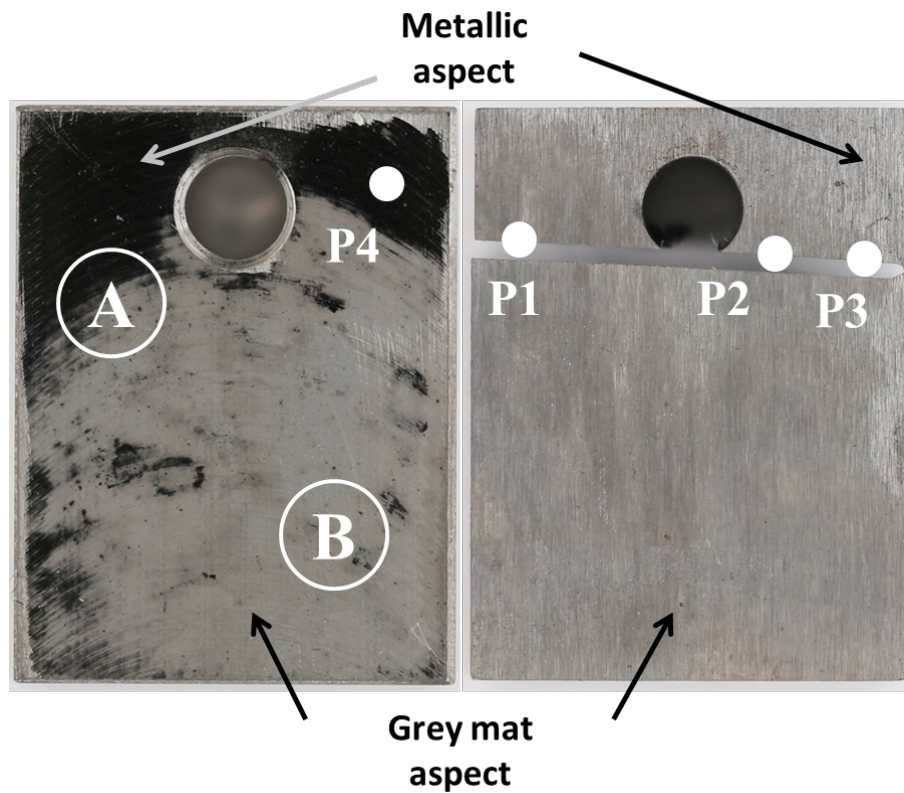


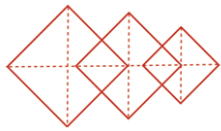
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a. Influence of water-dissolved oil contaminations

i. Oil 1 vol.%

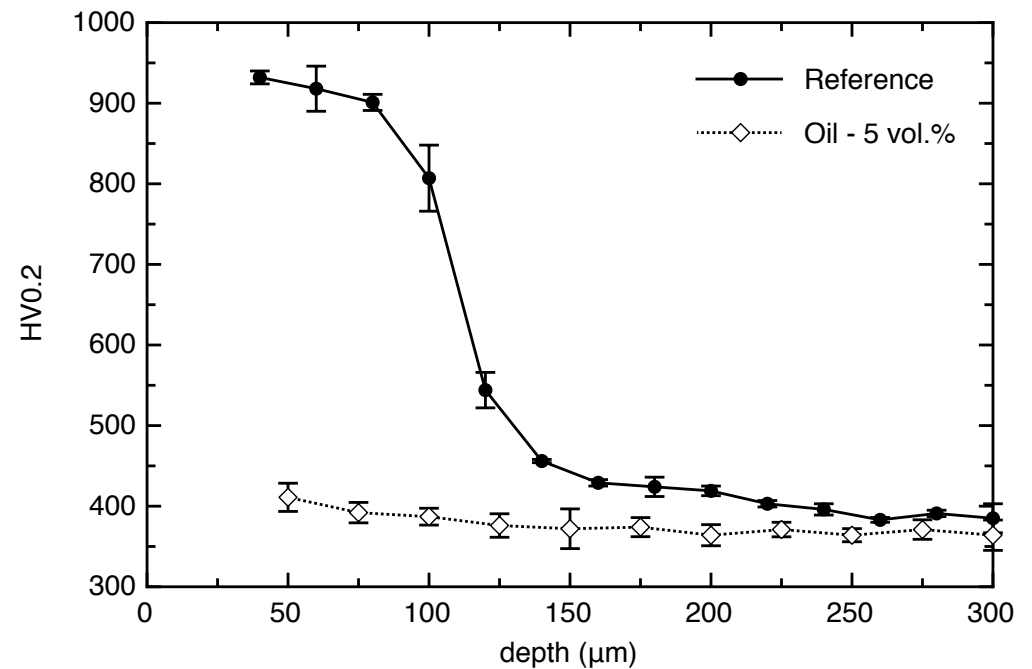
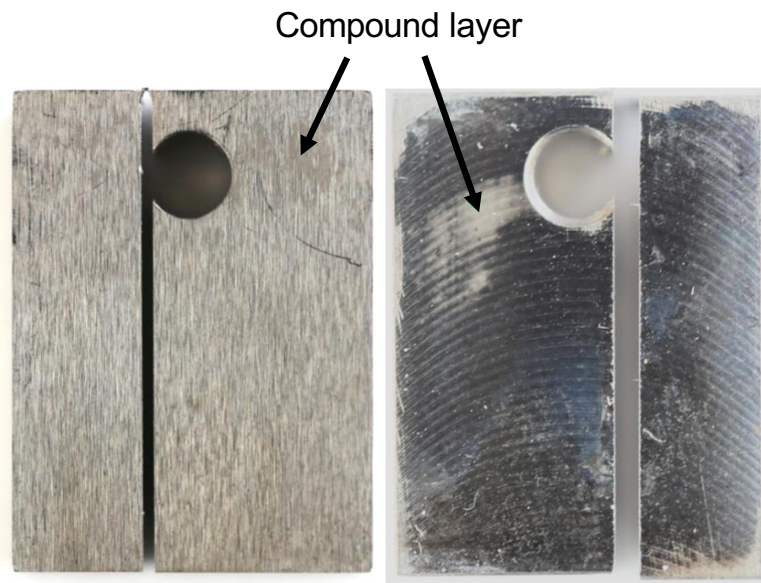


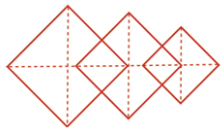


3. Results

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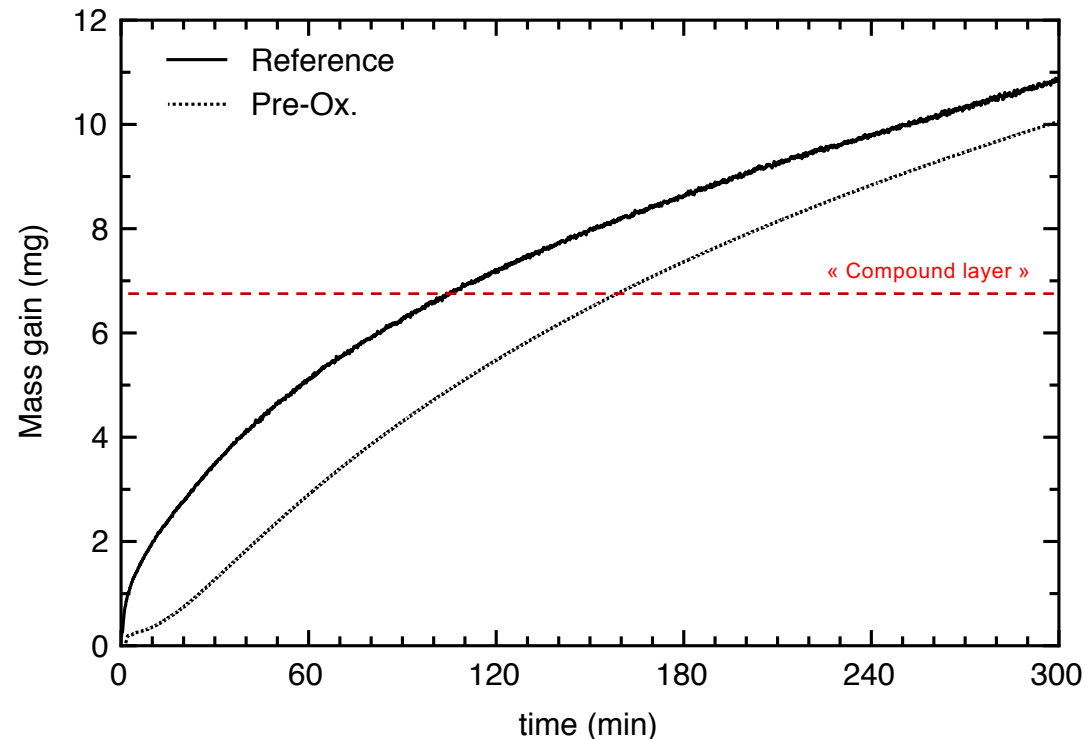
ii. Oil 5 vol.%

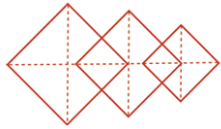




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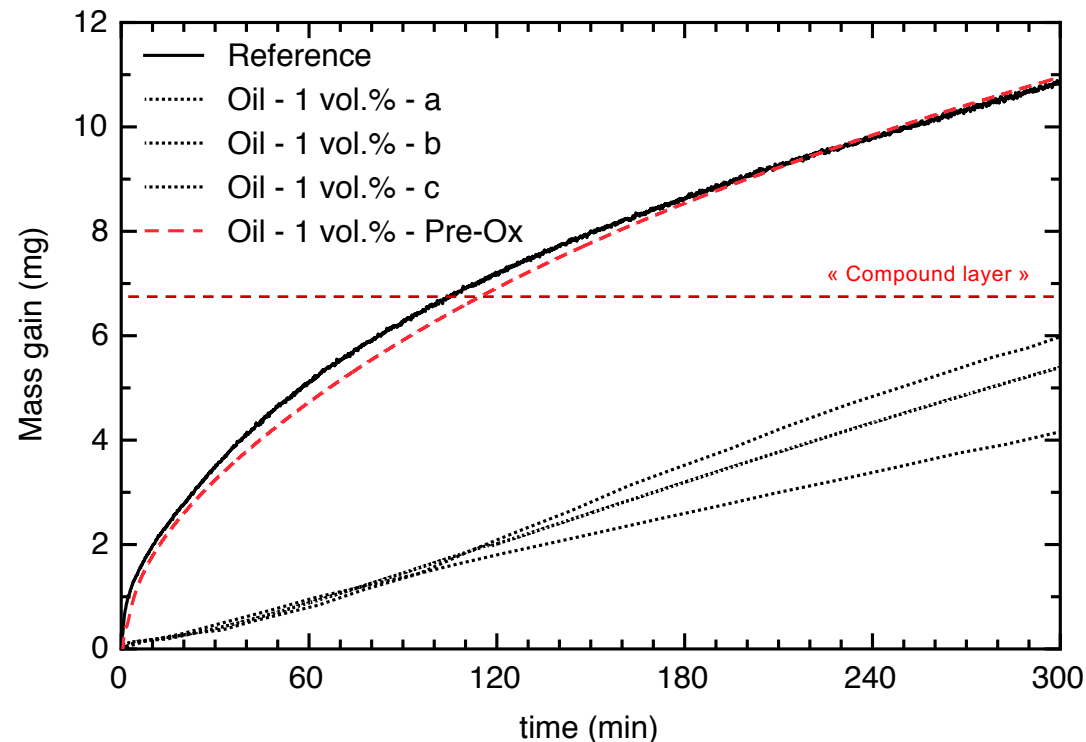
b. Influence of pre-oxidization (O_2 , 350 °C, 1h)

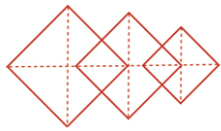




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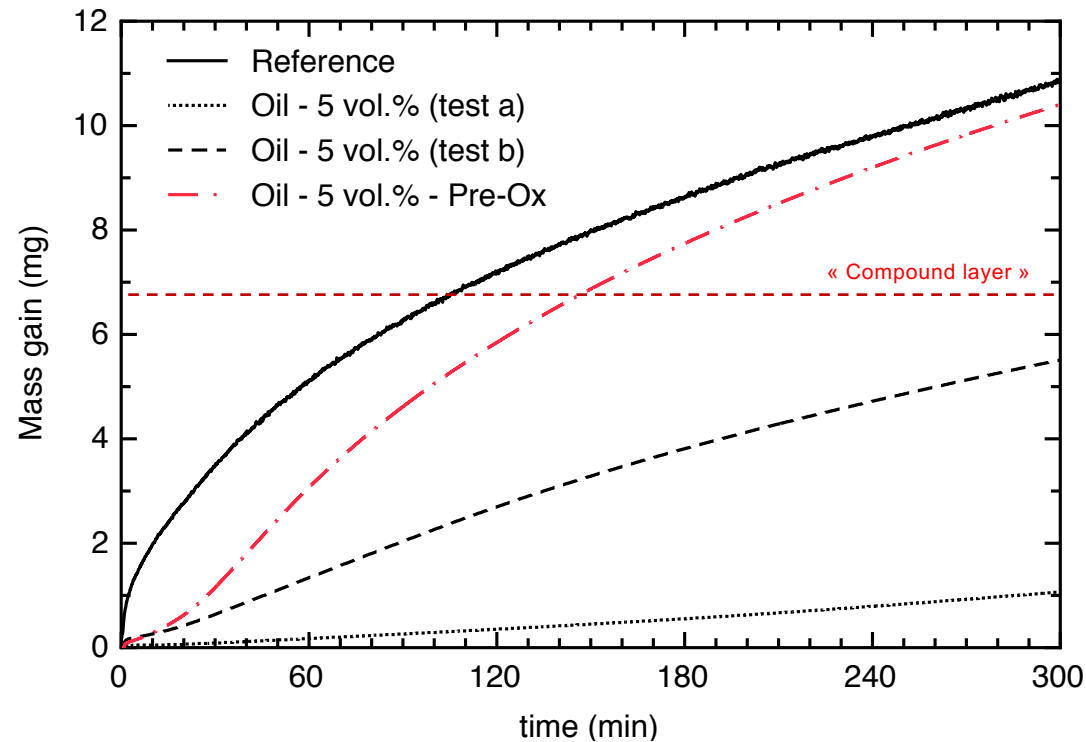
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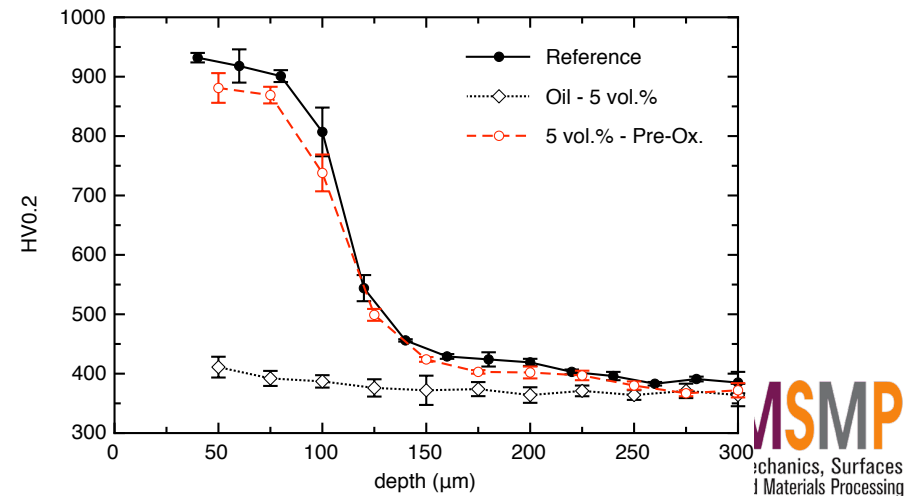
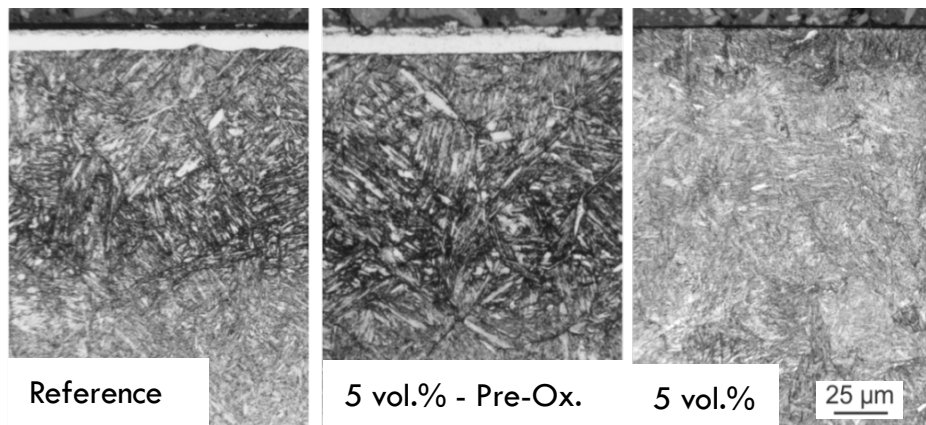
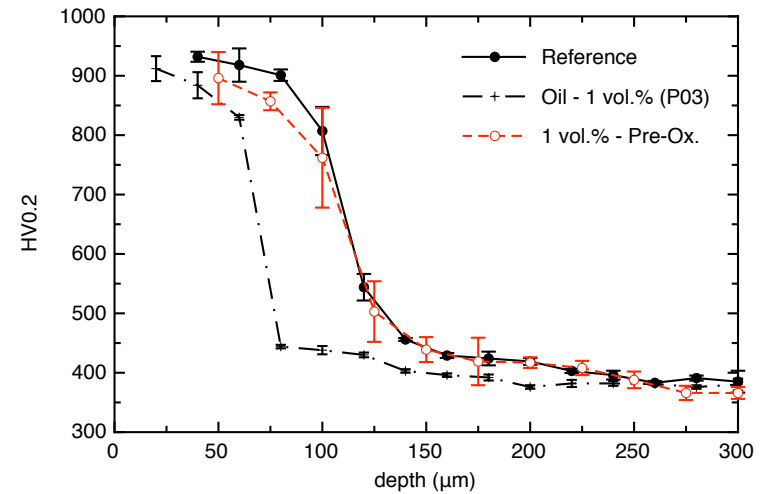
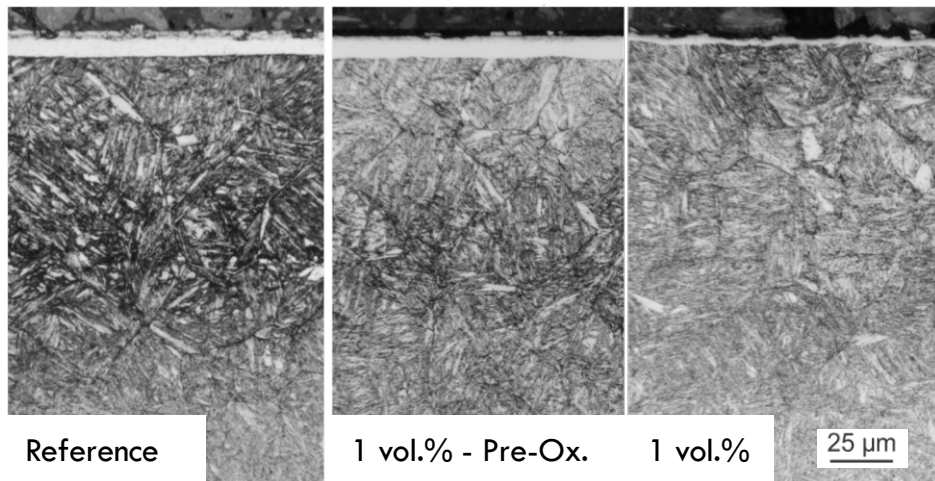
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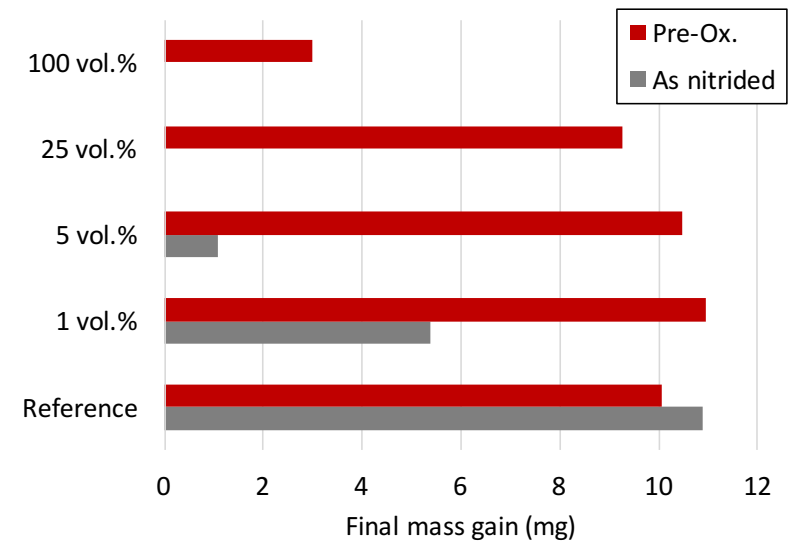
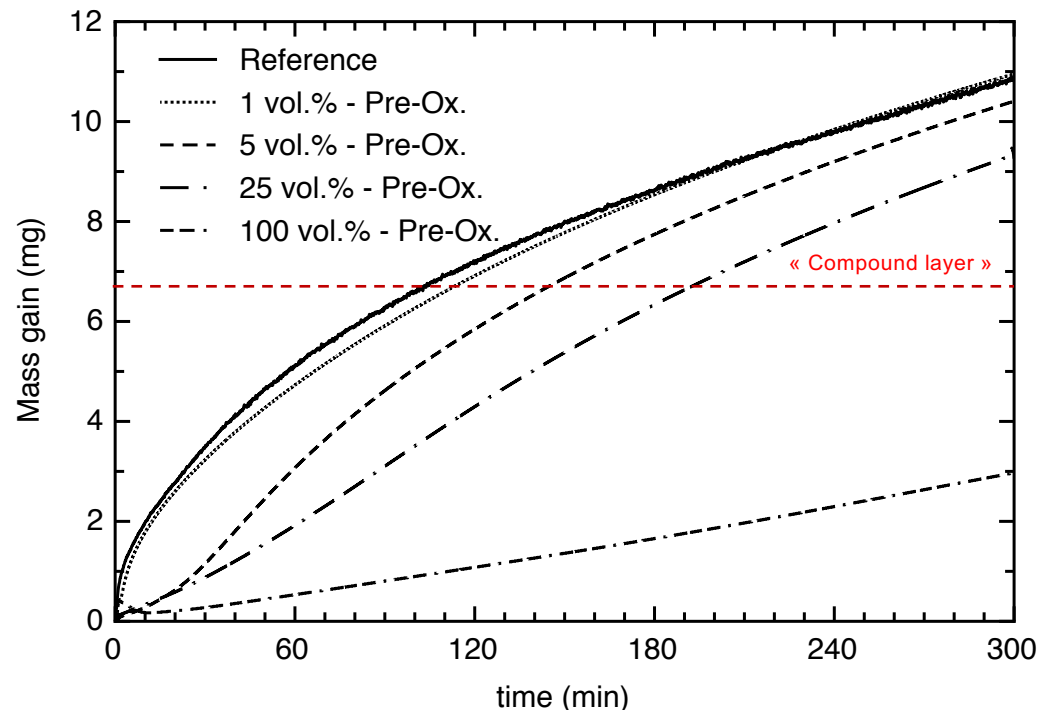
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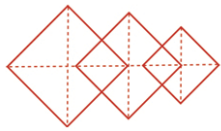
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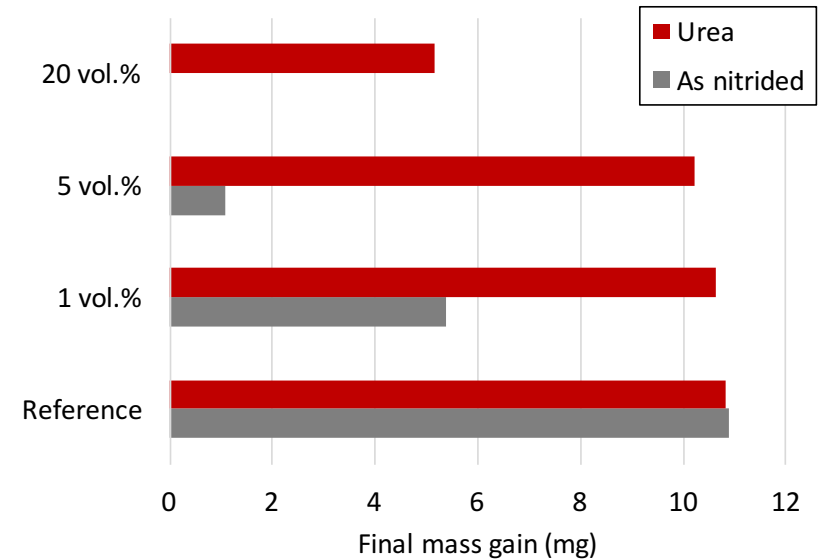
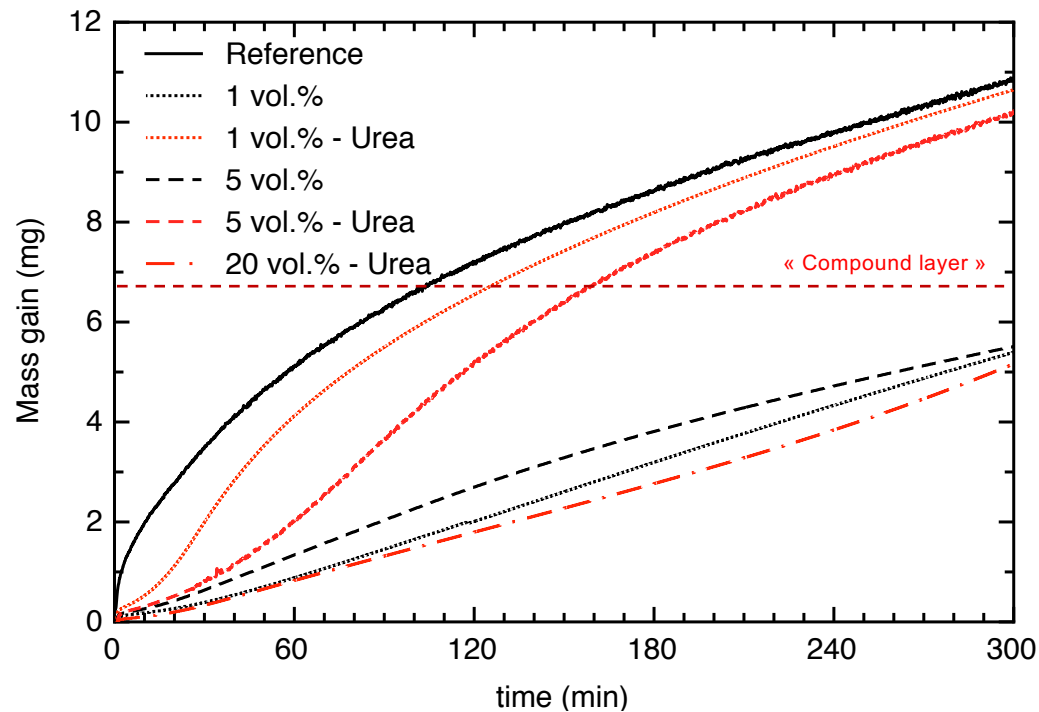
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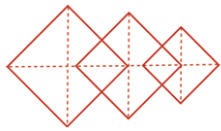




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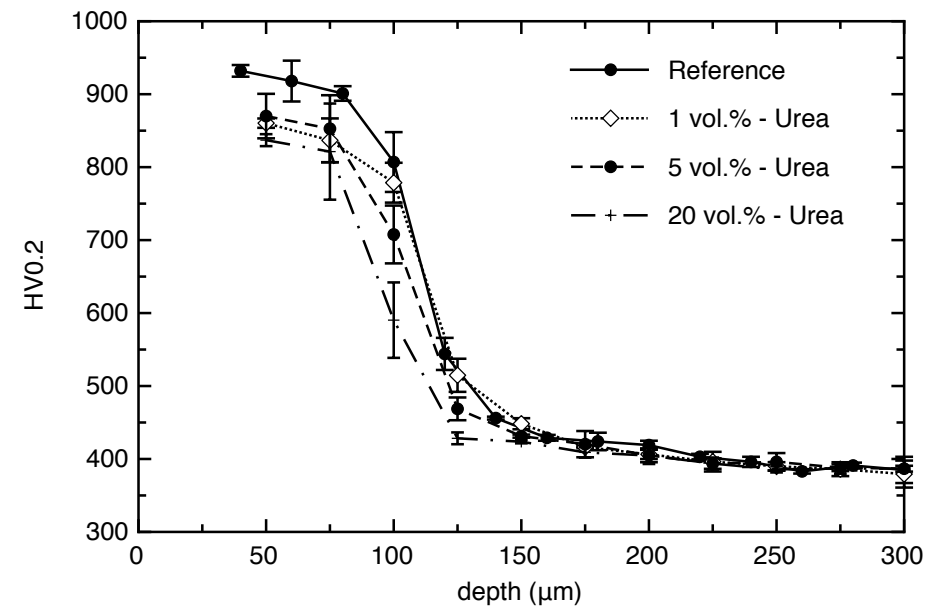
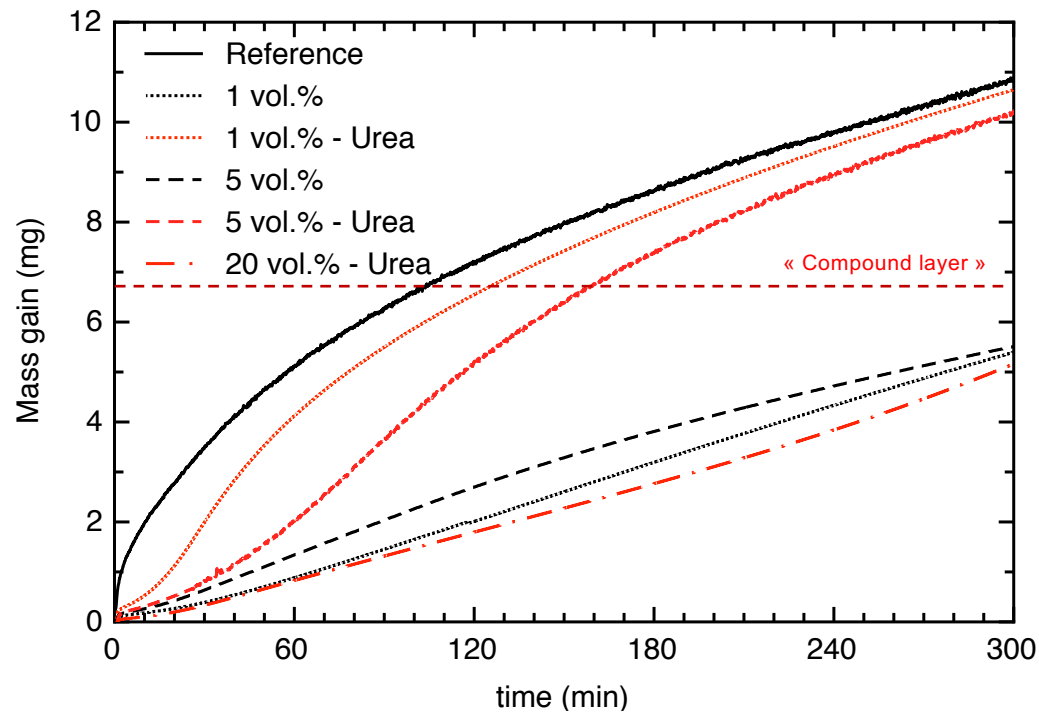
c. Influence of urea

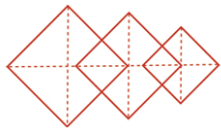




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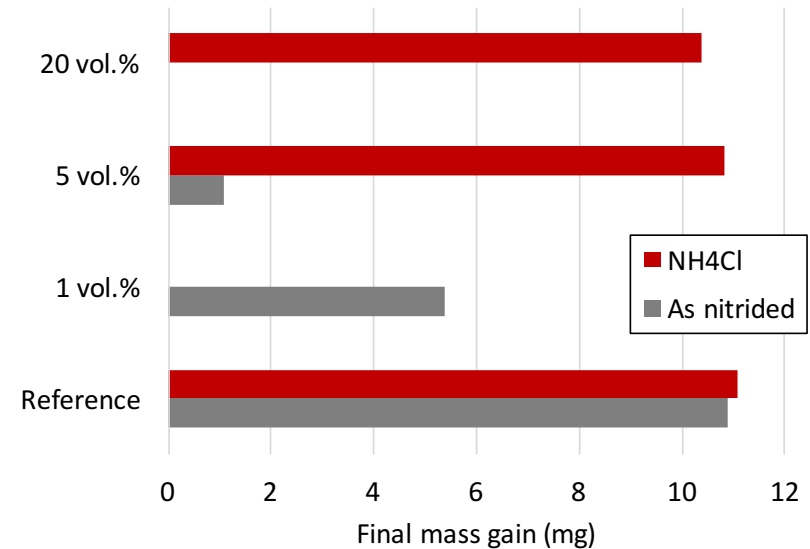
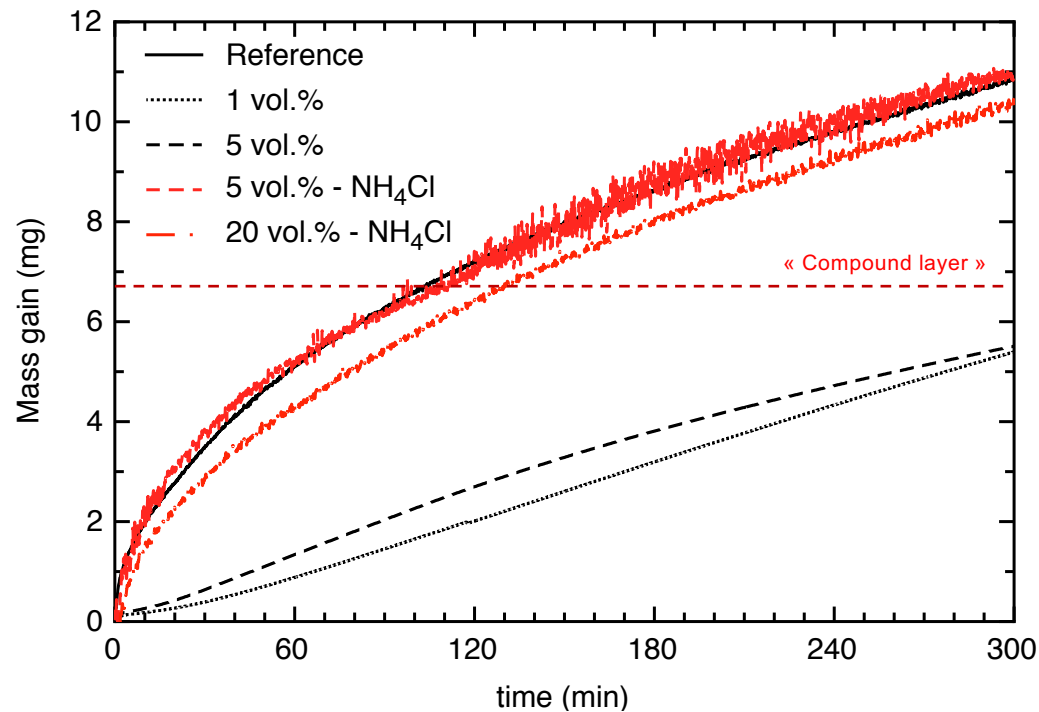
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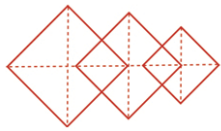




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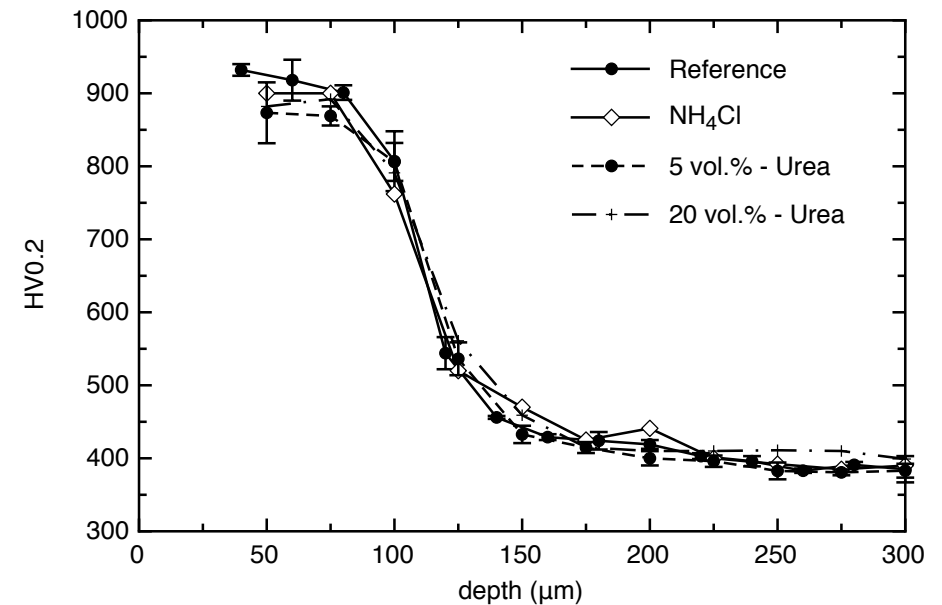
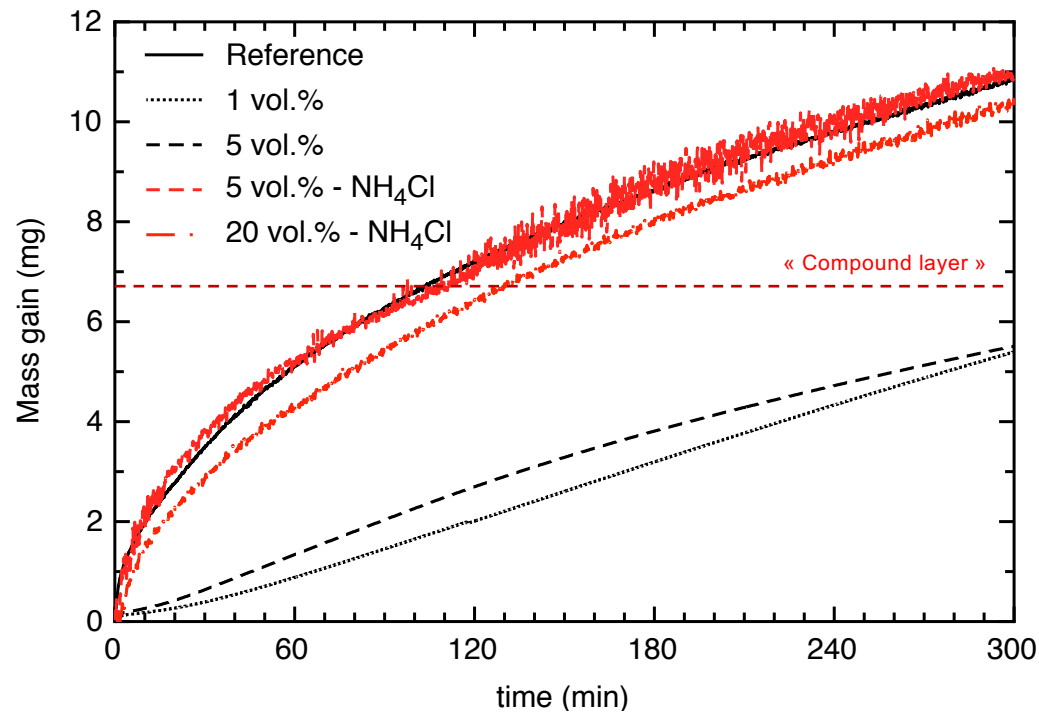
d. Influence of NH_4Cl

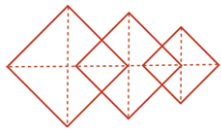




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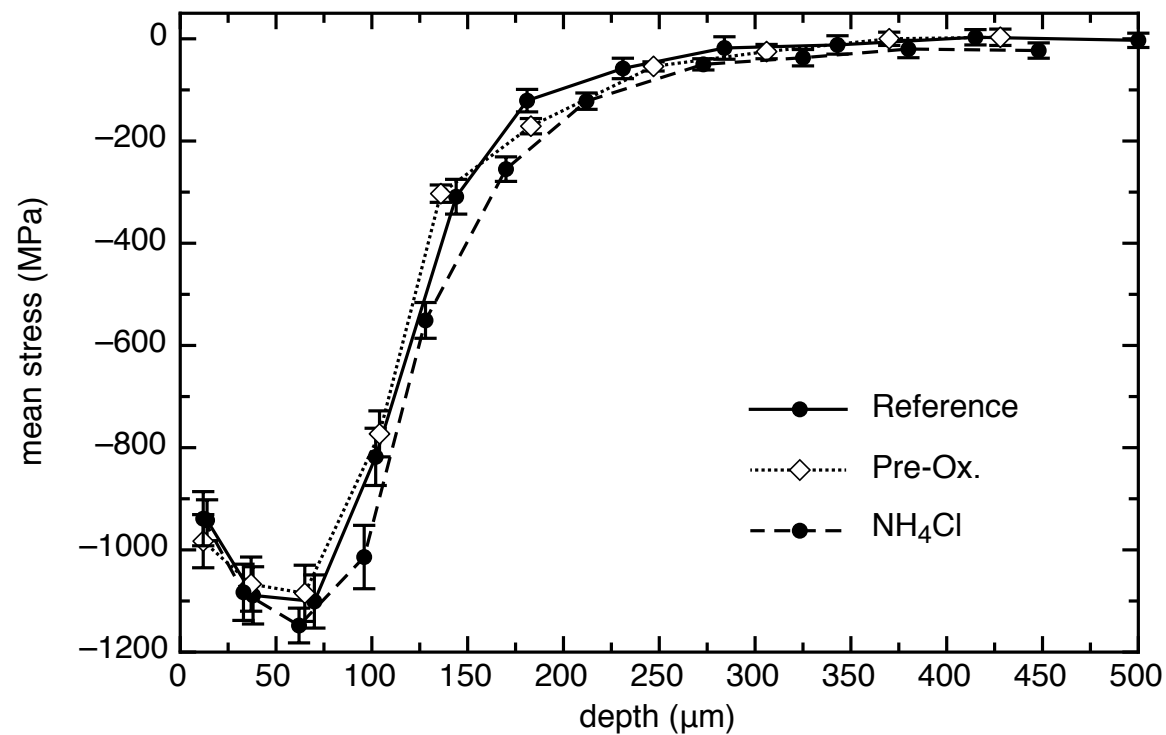
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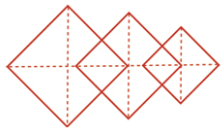




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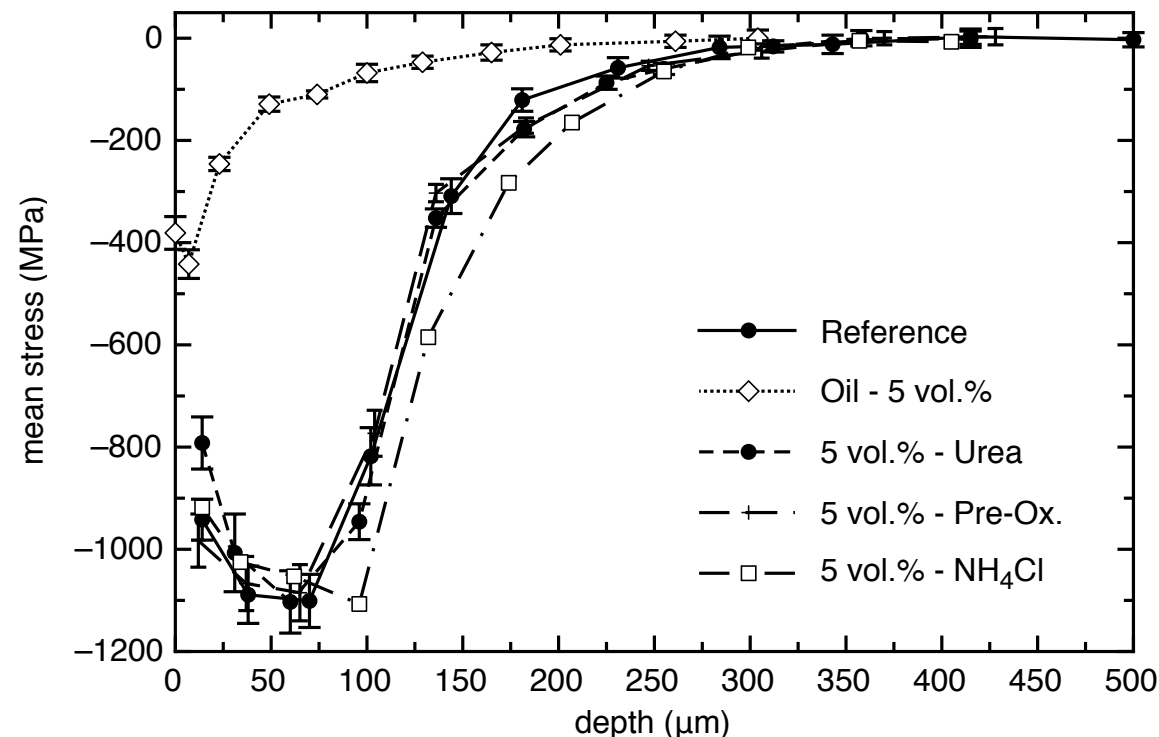
e. Pre-treatments & residual stresses

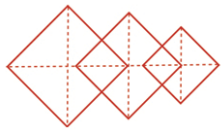




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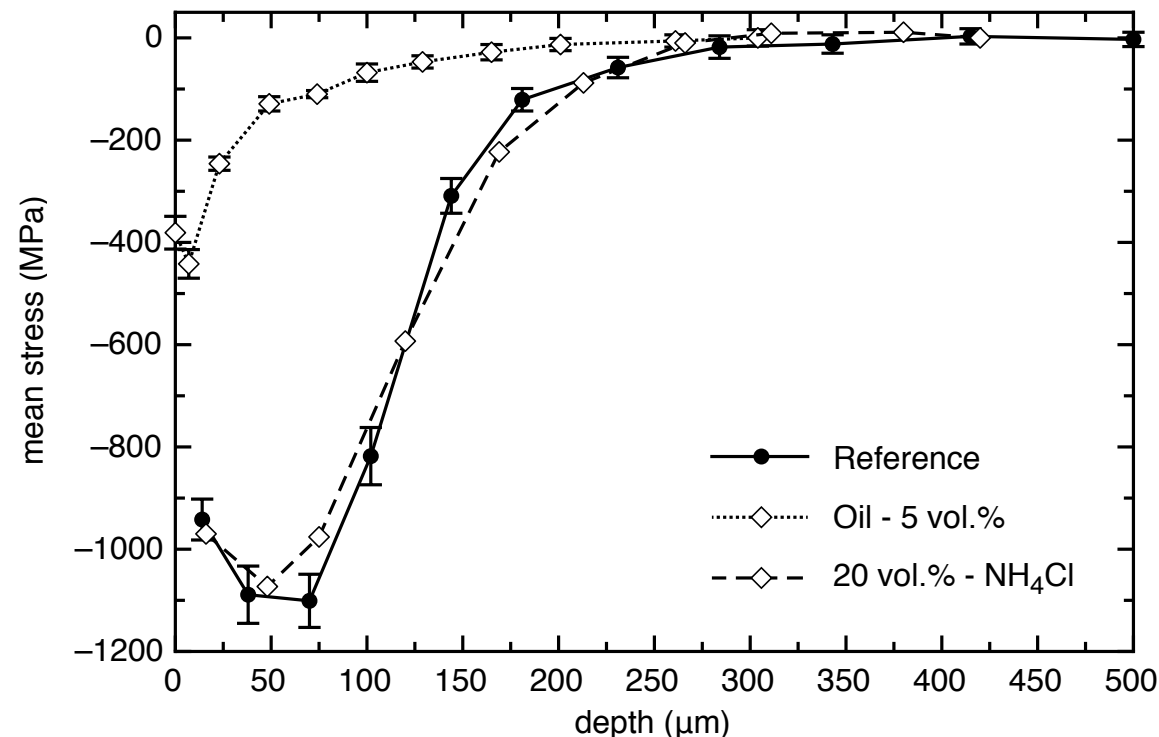
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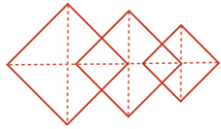




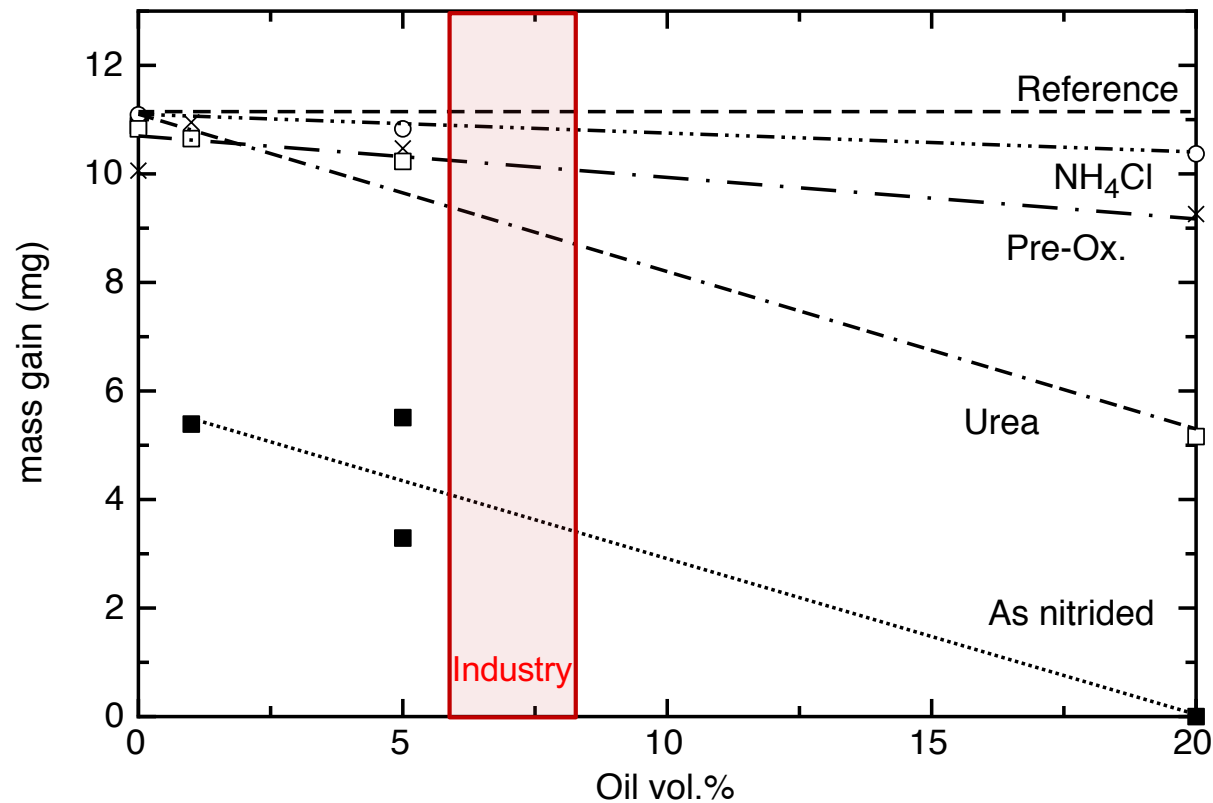
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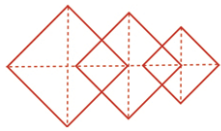
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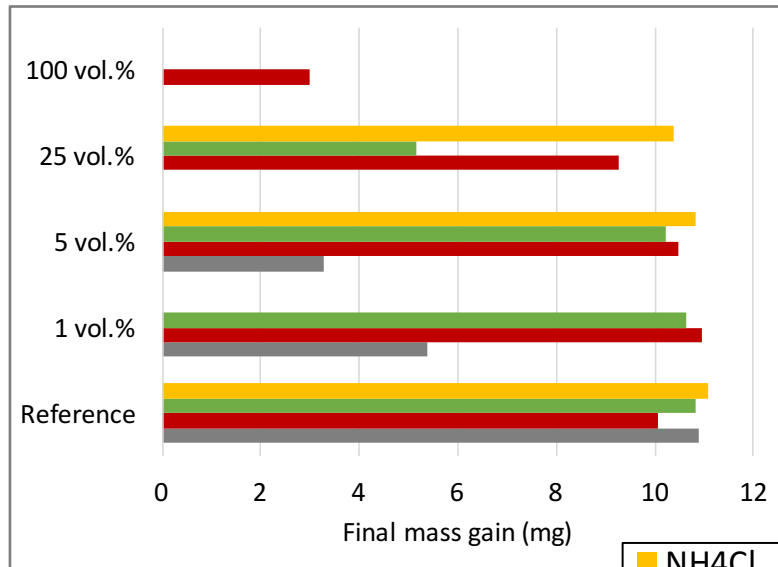


4. Conclusion (nitriding 520 °C, 5 h, K_N 3,7 atm^{-1/2})

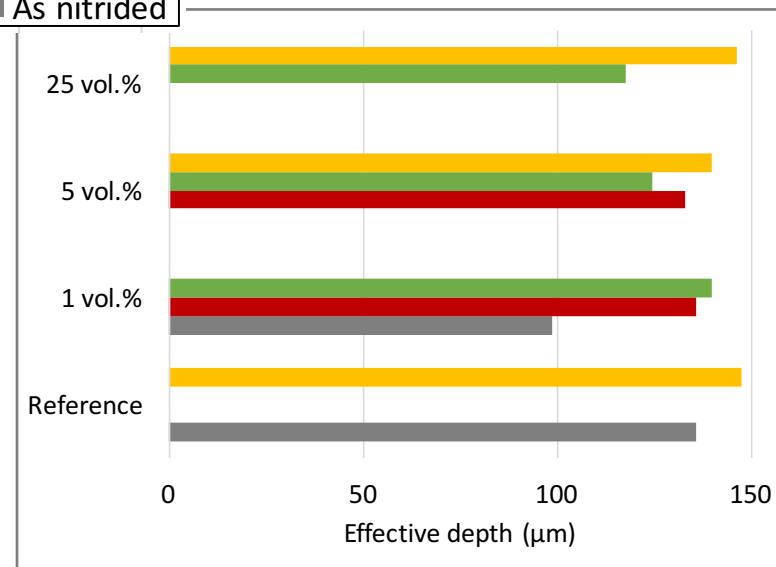
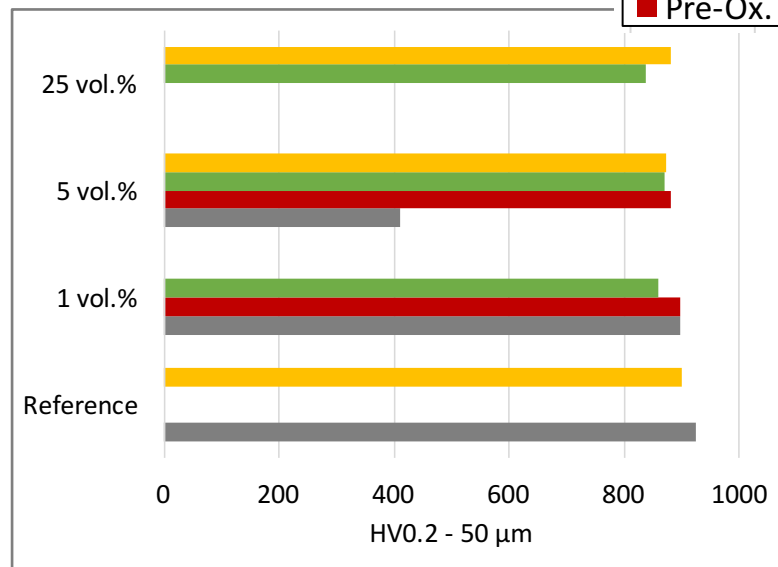




4. Conclusion (nitriding 520 °C, 5 h, K_N 3,7 atm^{-1/2})



- In-situ pre-treatment:
 - Urea
 - Oxidization (thickness layer dependence)
 - NH₄Cl
- Advantage:
 - In-situ (during the heating stage)
 - NH₄Cl
 - Decomposition into NH₃
 - Acidic cleaning/sanding



*Thank you for your
attention !*

B.Guillot, S.Jégou, L.Barrallier, *Degradation of gaseous nitriding of steel by lubricant contamination - Effect of in-situ pre-treatments*, submitted to Surface & Coating Technology (2016)