



Arc et Senans, 21-24 Nov 2002



VPO catalysts for the oxidation of n-butane to MA

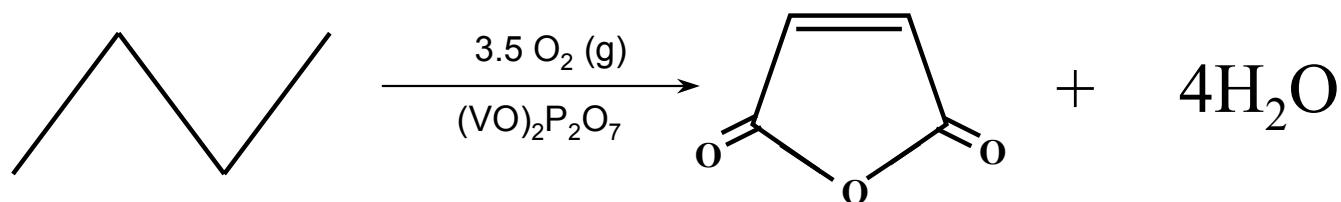
ANDRZEJ LISKOWSKI



Introduction



Oxidation of n-butane to maleic anhydride



APPLICATIONS

unsaturated polyester resins

Fiberglass composites

Lube oil

Fumaric acid

Assorted polymers



Microstructure characterization



microstructure

Set of samples

- single crystal of VPO
- standard phases, e.g. $\alpha_1\text{-VOPO}_4$
 - CAT series
- VPA, VPD, VPO series

XRD analysis

- phase analysis

SEM characterization

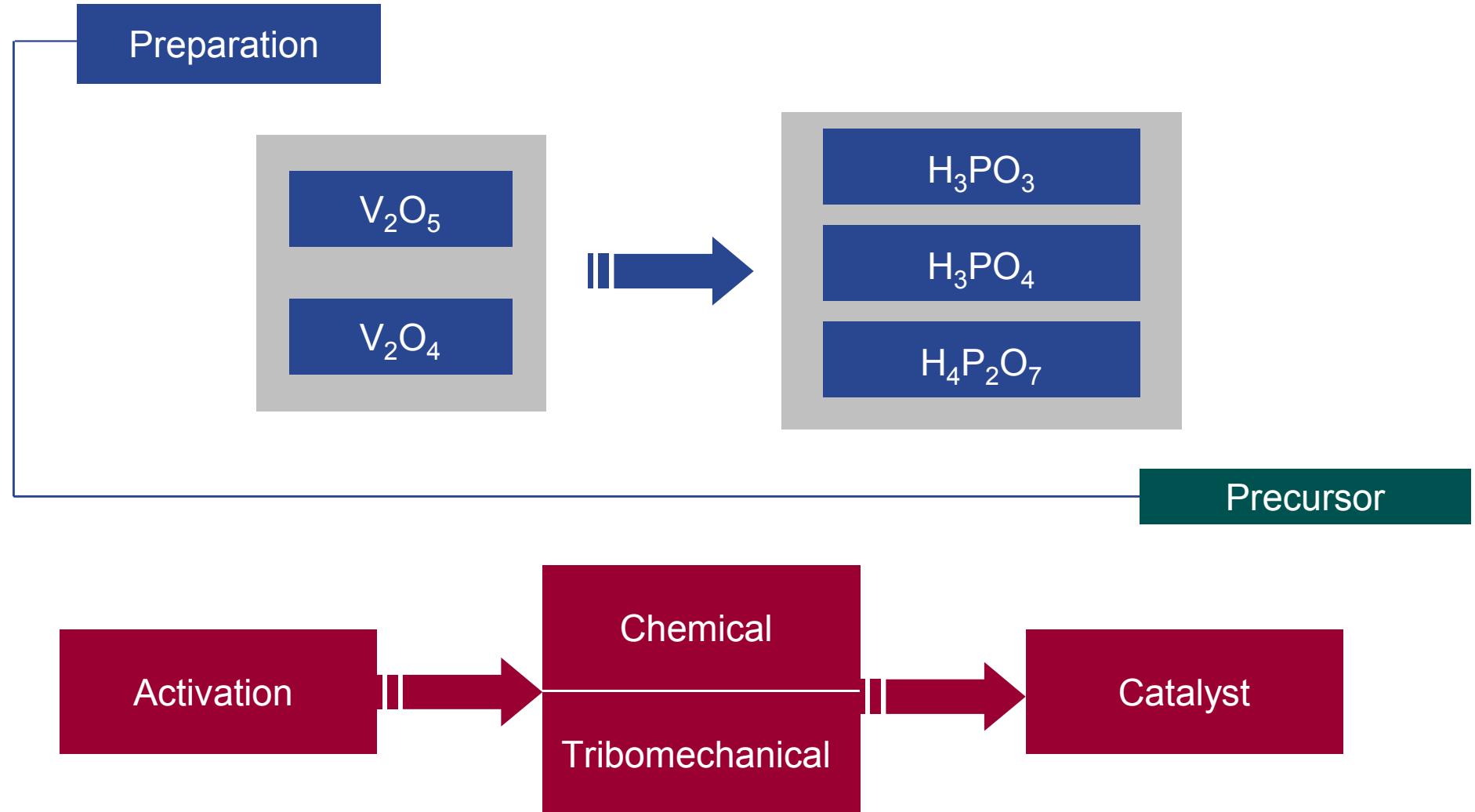
- morphology
- chemical composition

TEM

- LM images
- HREM images
- diffraction patterns
- EELS



VPO samples





After Activation



Chemical activation

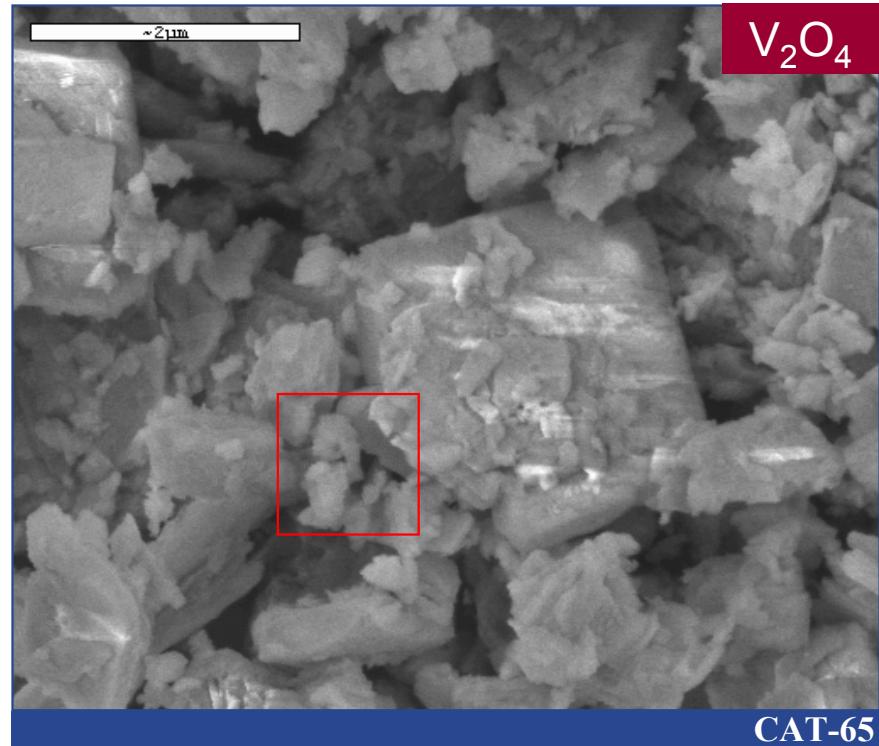
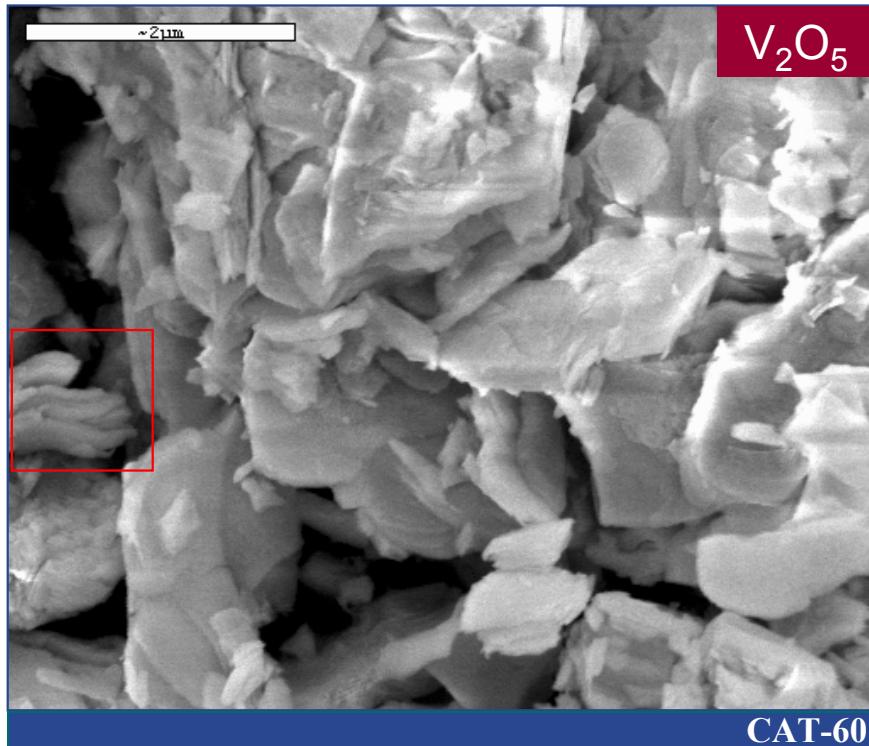
(VO) ₂ P ₂ O ₇
α_I -VOPO ₄
α_{II} -VOPO ₄
β -VOPO ₄ ?
δ -VOPO ₄ unknown
γ -VOPO ₄ unknown

Tribomechanical activation

(VO) ₂ P ₂ O ₇
Multiplicity of phases

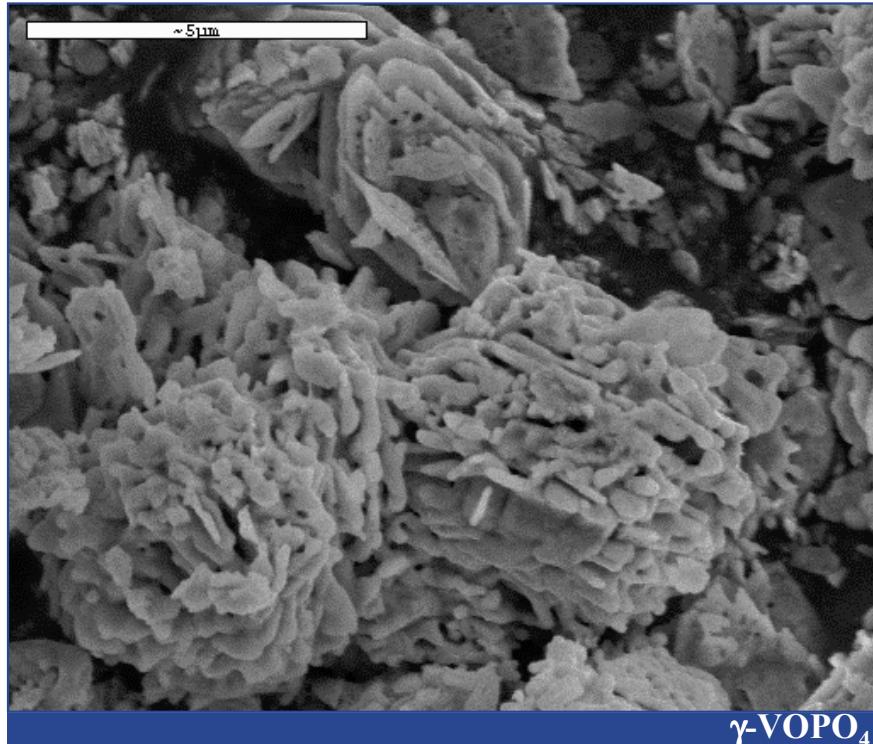
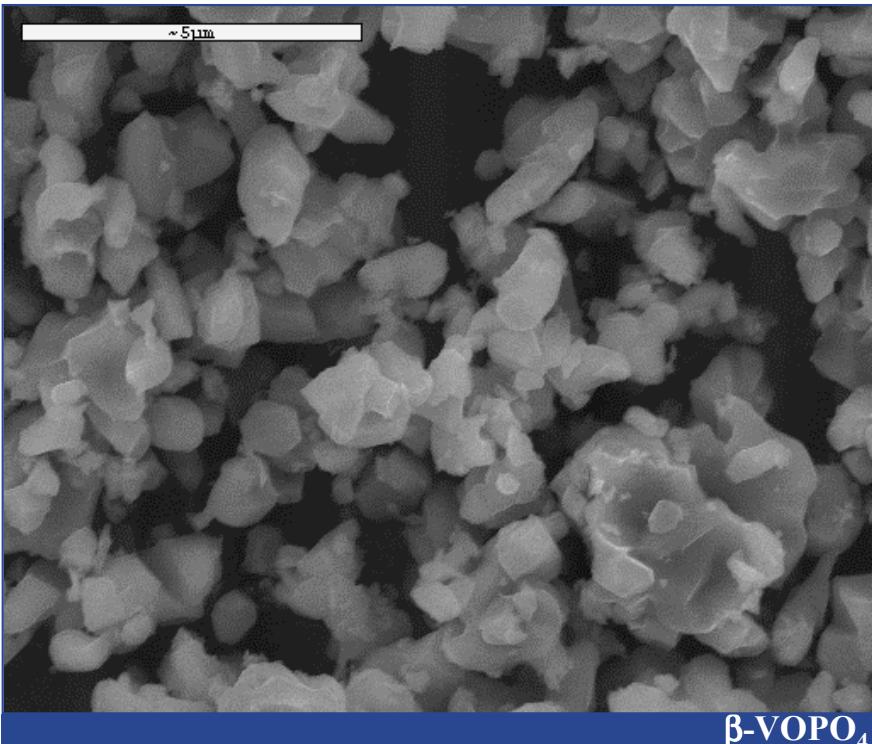


Morphology – SEM images



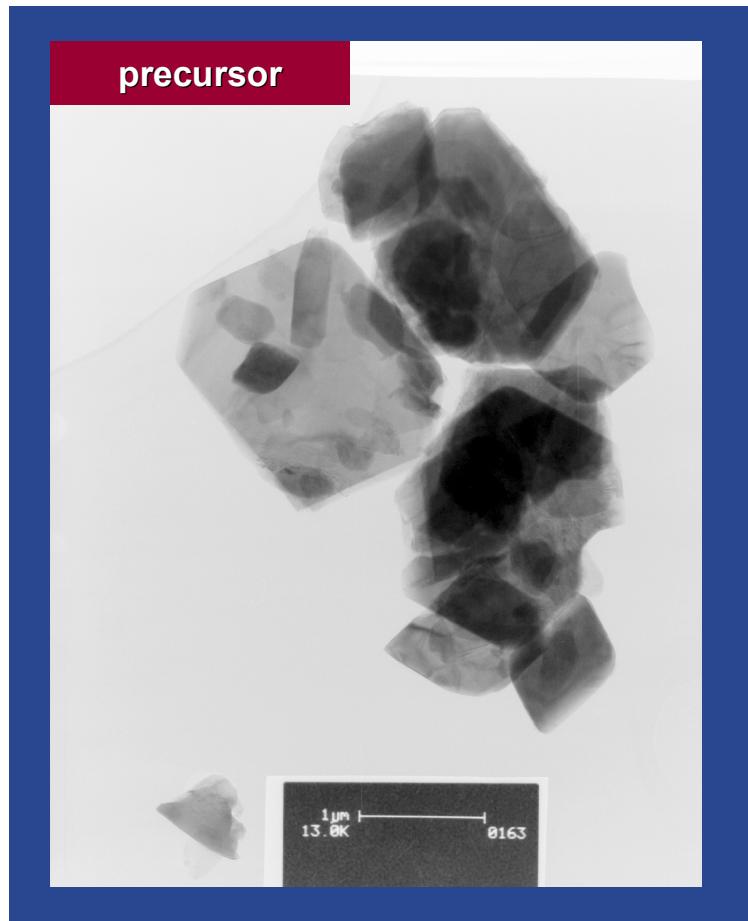


Standard phases: β -, γ -VOPO₄

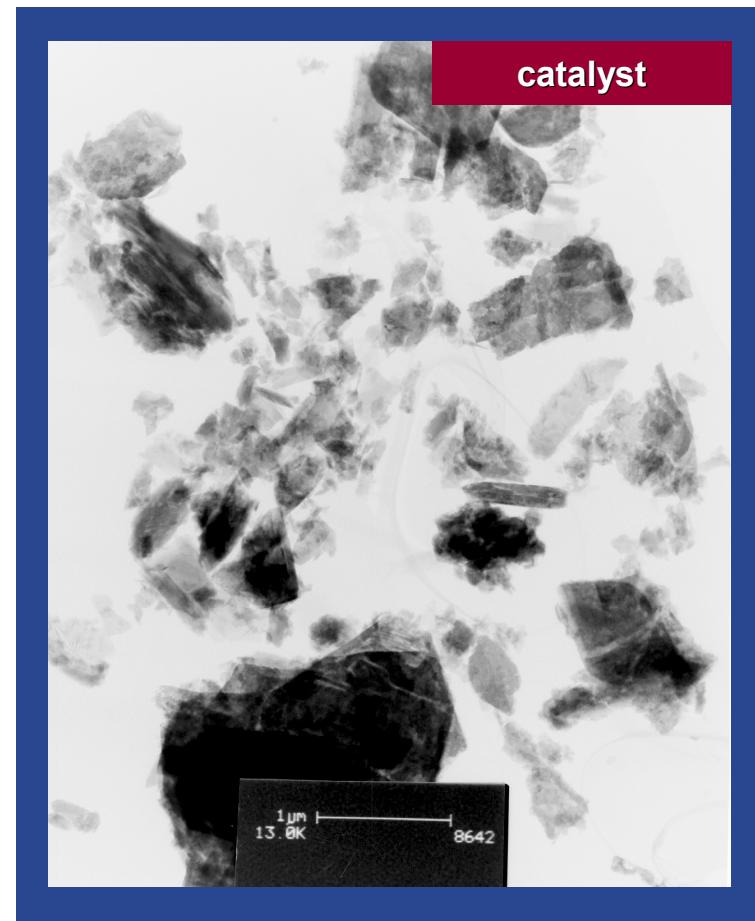




Structure evaluation - DP



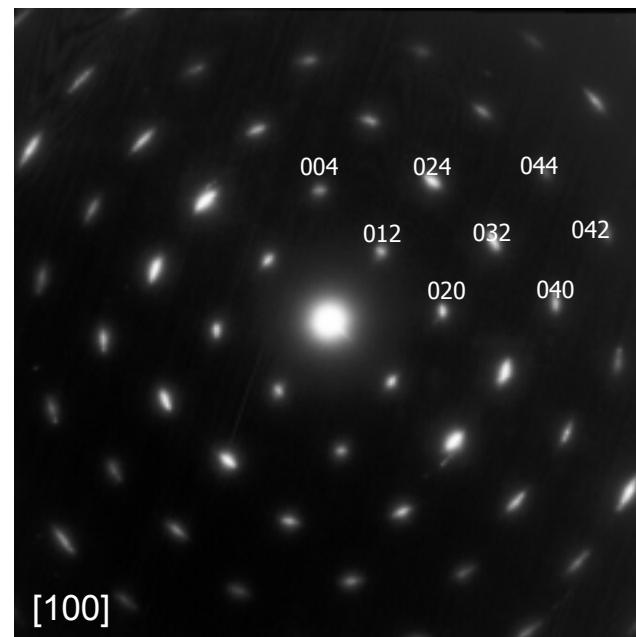
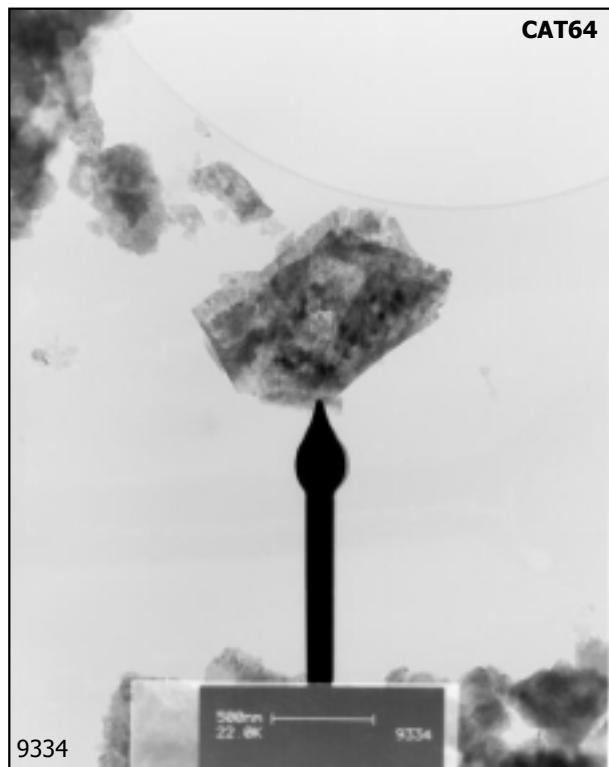
$\text{VOHPO}_4 \cdot 0.5\text{H}_2\text{O}$



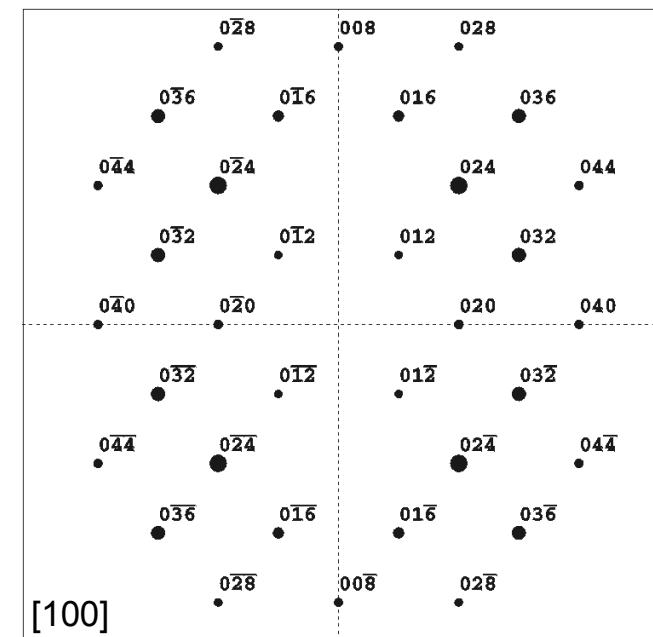
$(\text{VO})_2\text{P}_2\text{O}_7$, V^{5+} phases



Closer look - DP



Experimental DP



Simulated DP

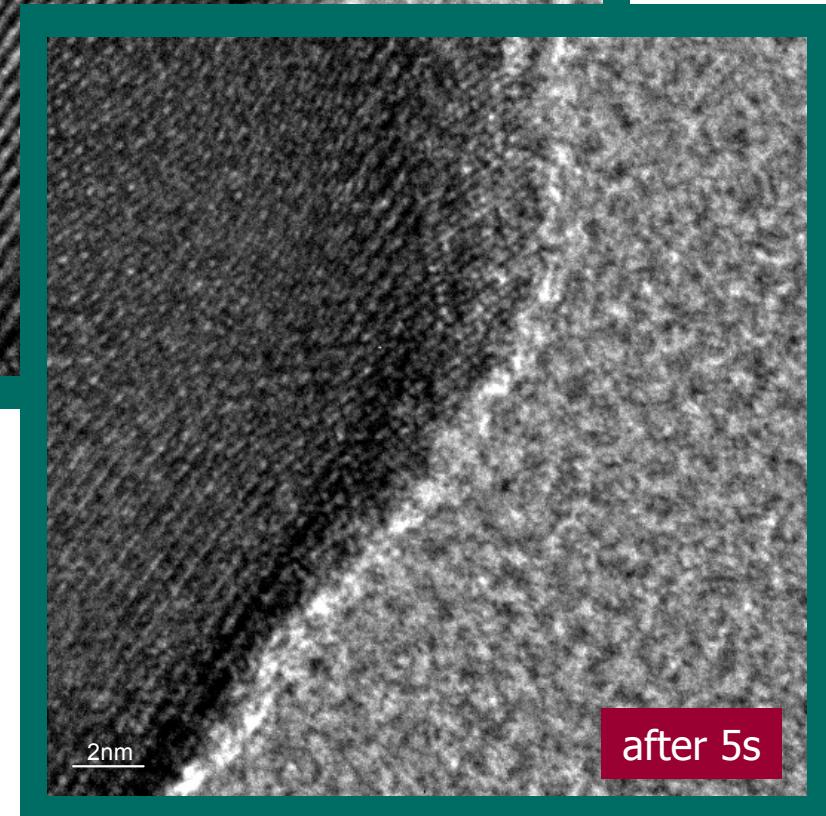
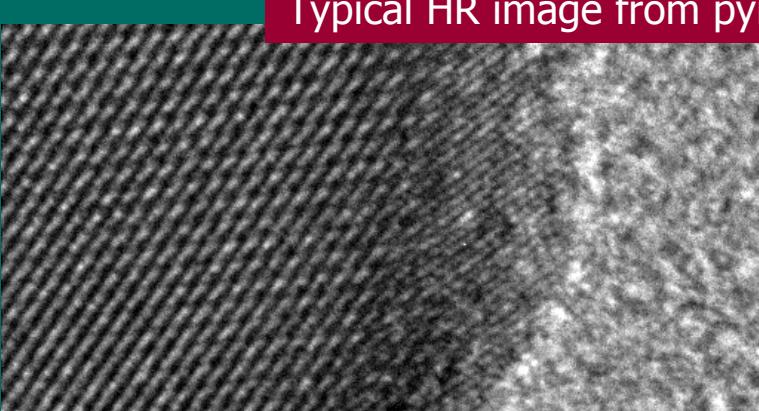
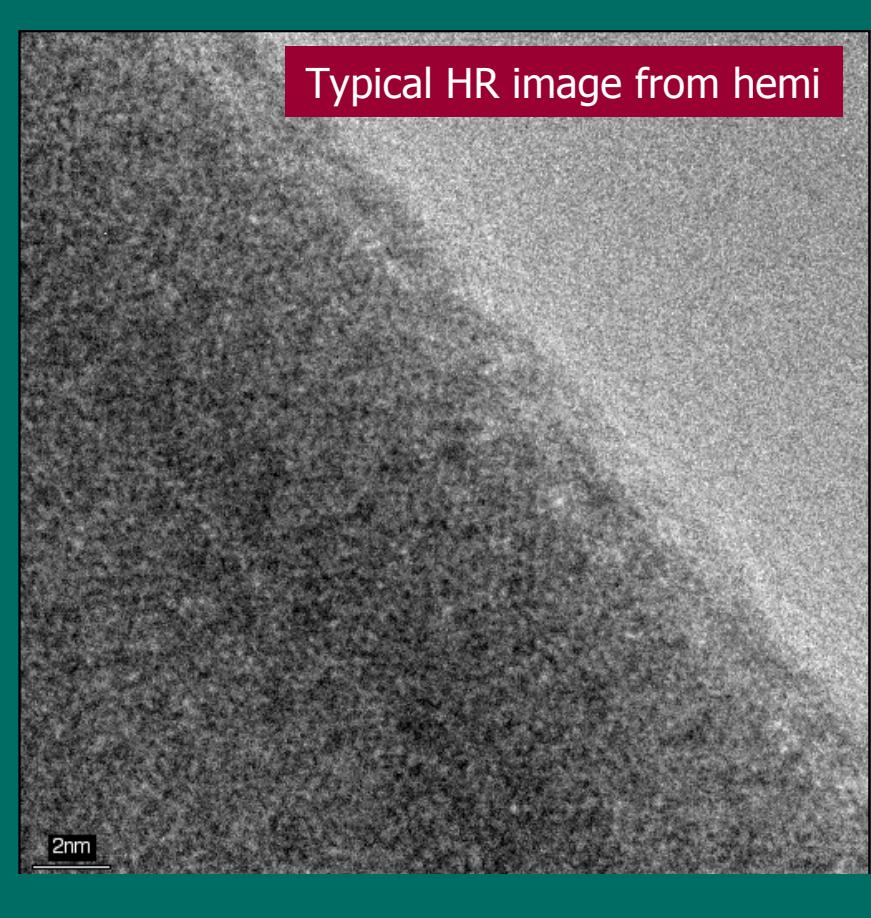


HR images: hemihydrate and pyrophosphate



Typical HR image from pyro

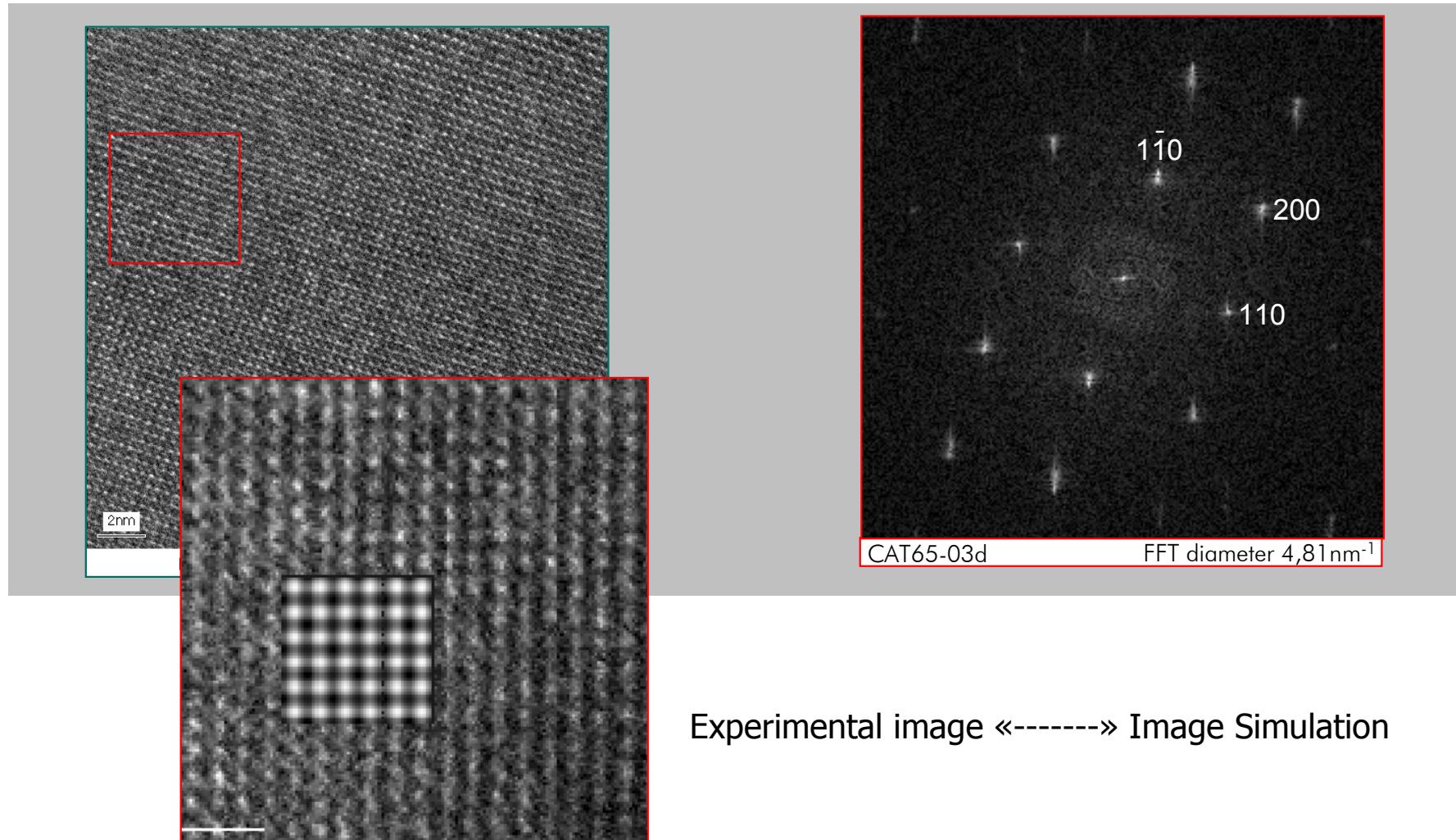
Typical HR image from hemi



after 5s

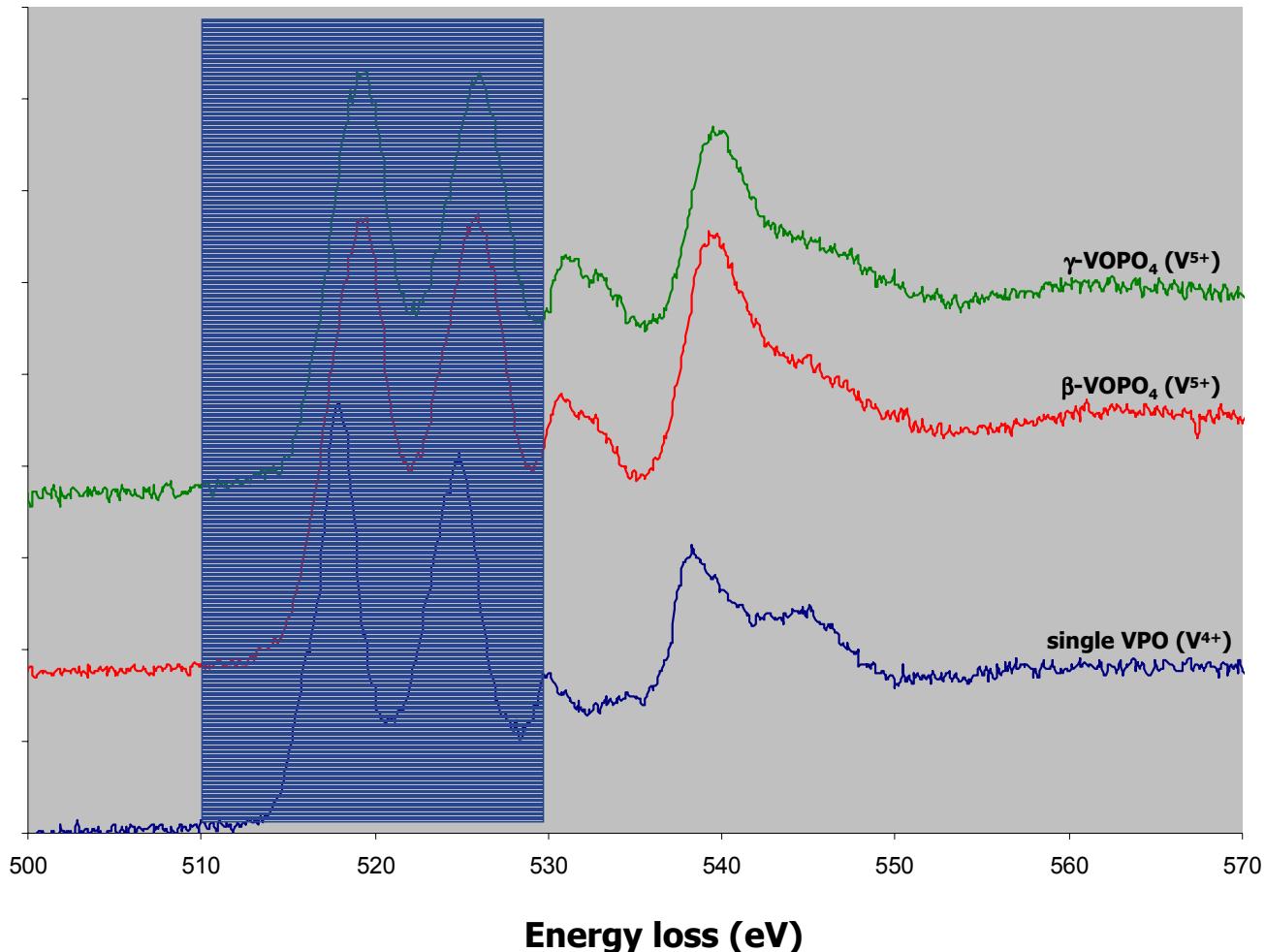


Microstructure - $\alpha_1\text{-VOPO}_4$



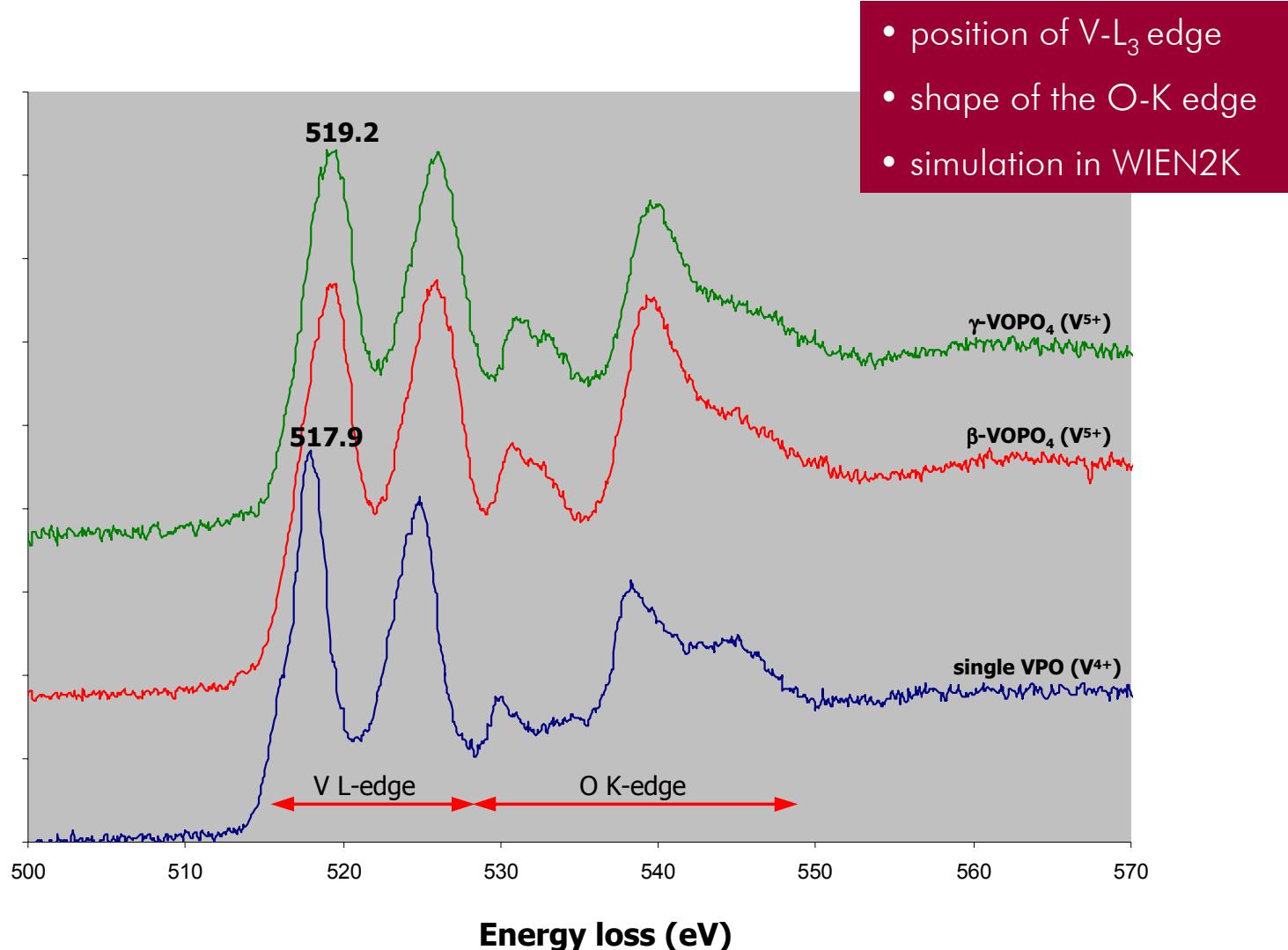


Electron Energy Loss Spectrum



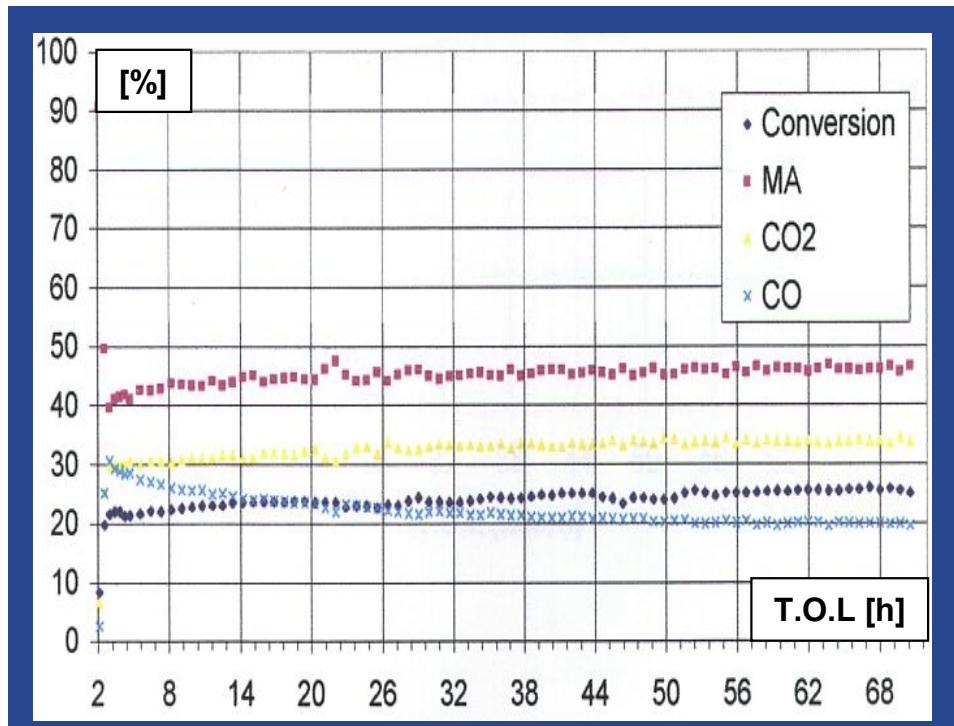


Electron Energy Loss Spectrum

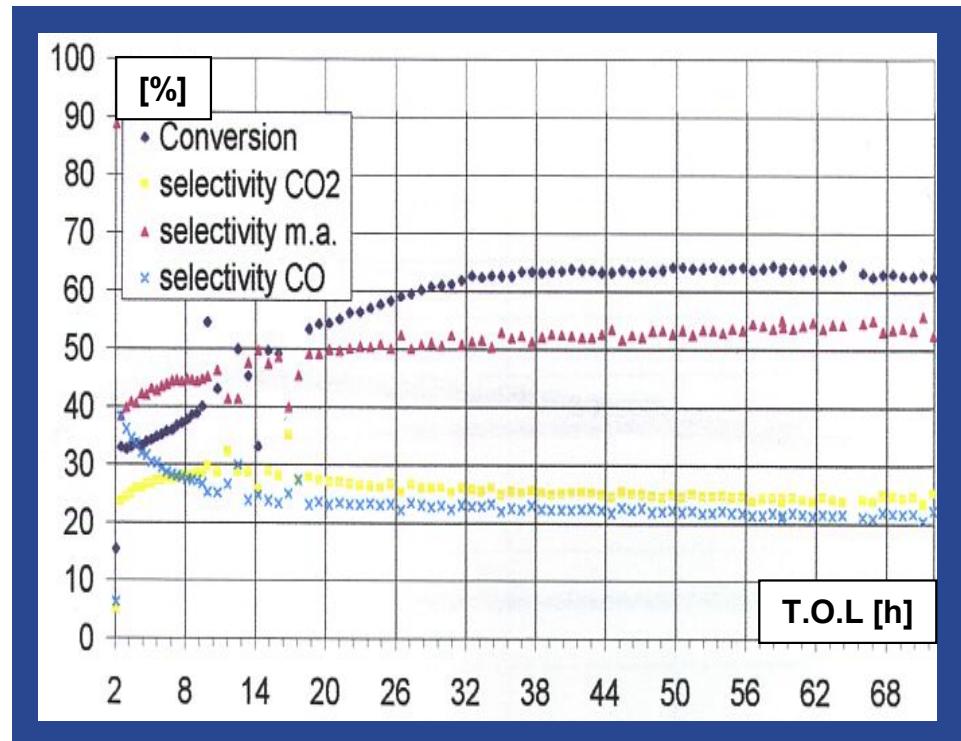




Catalytic performance



cat60



cat61

Microstructural changes after refluxing?



Thank you!

