UNIVERSITEIT GENT

FACULTY OF **ENGINEERING AND ARCHITECTURE**

Fe_2O_3-CeO_2 for Chemical looping process: in situ XRD and XAS study

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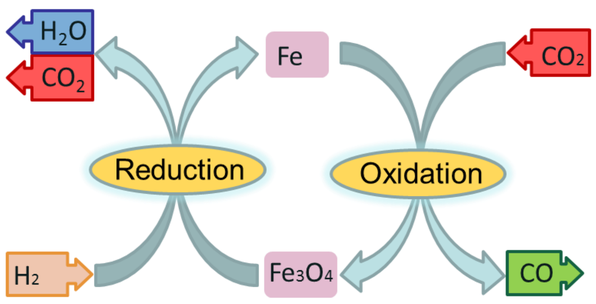
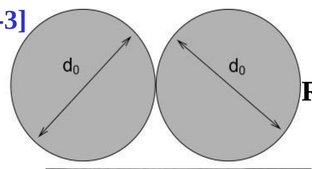
Belgian Science Policy Office belspo

ERIC European Research Institute of Catalysis

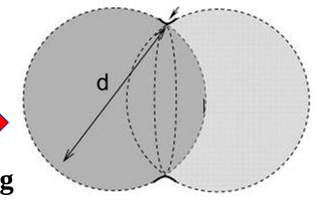
Introduction: Chemical Looping[1-3]

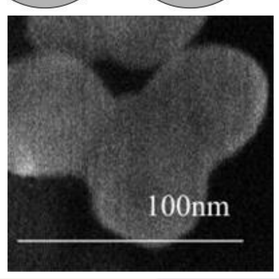


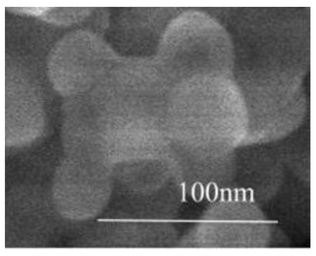


Repeated cycling

Sintering

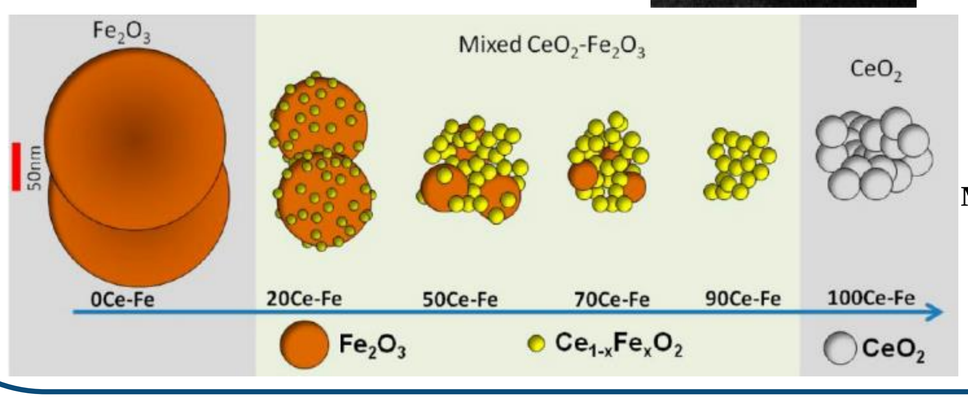




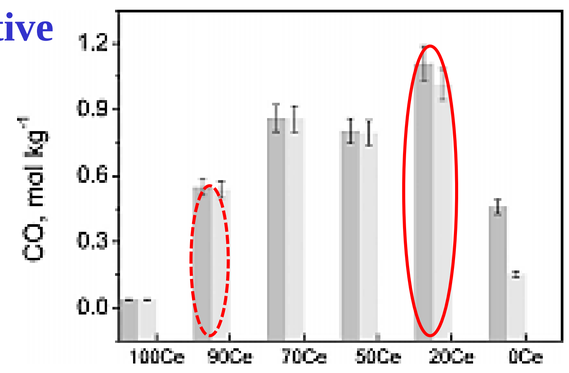


Prevent sintering

Modify iron oxides

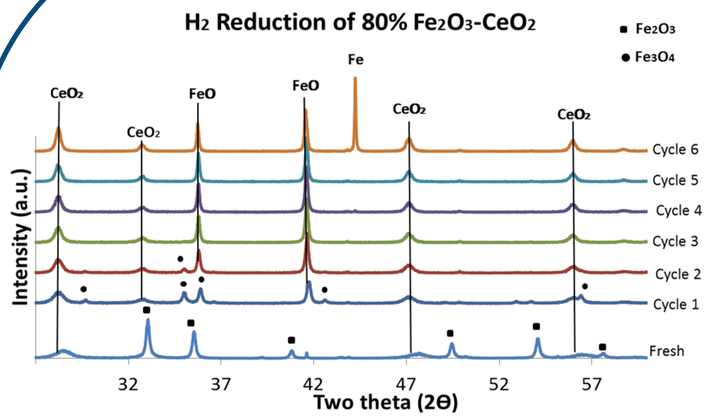


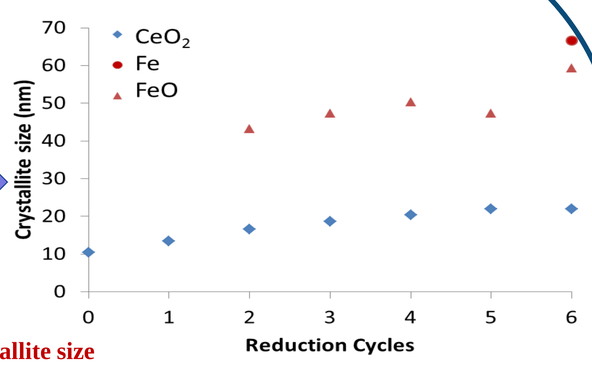
Objective



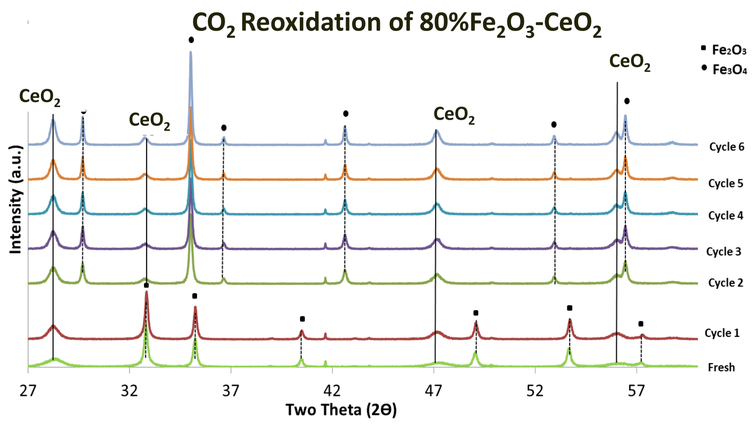
In order to understand the structural interaction, a series of Fe_2O_3 was prepared and investigated using in situ XRD and XAS at 700°C in chemical looping process. The most active material (80% Fe_2O_3-CeO_2) from our previous study [1] and the material with a low amount of incorporated Fe (10% Fe_2O_3-CeO_2) as seen from the change in lattice parameters, have been pursued for this investigation.

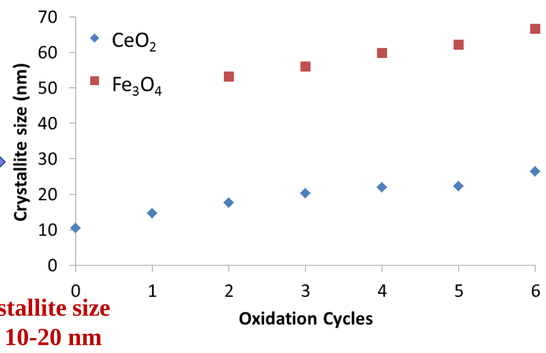
In situ XRD study



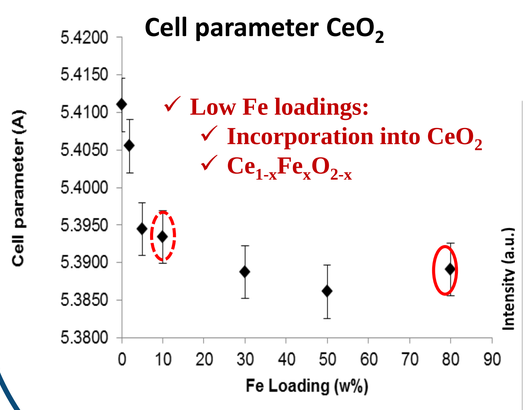


- ✓ **Stable crystallite size**
  - ✓ **CeO_2 : 10-20 nm**
  - ✓ **FeO : 40-55 nm**
  - ✓ **Fe : 60 nm**
- ✓ **Sequence of reduction: Fe_2O_3 → Fe_3O_4 → FeO→ Fe**

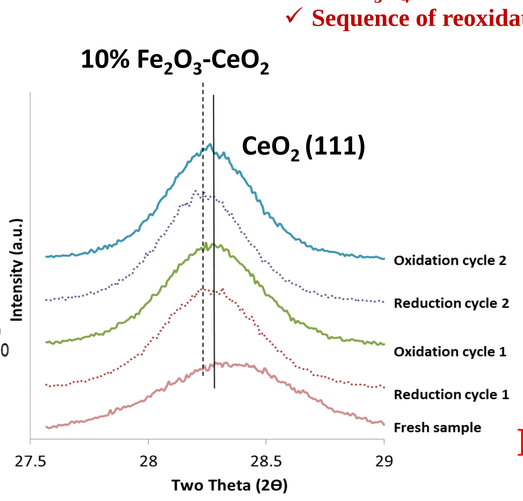




- ✓ **Stable crystallite size**
  - ✓ **CeO_2 : 10-20 nm**
  - ✓ **Fe_3O_4 : 50-60 nm**
- ✓ **Sequence of reoxidation: Fe→Fe_3O_4**



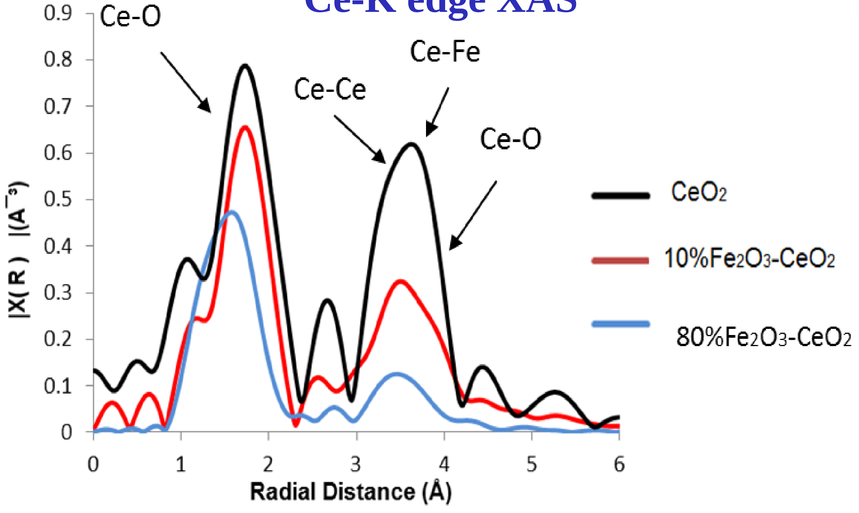
- ✓ **Low Fe loadings:**
  - ✓ **Incorporation into CeO_2**
  - ✓ **$Ce_{1-x}Fe_xO_{2-x}$**



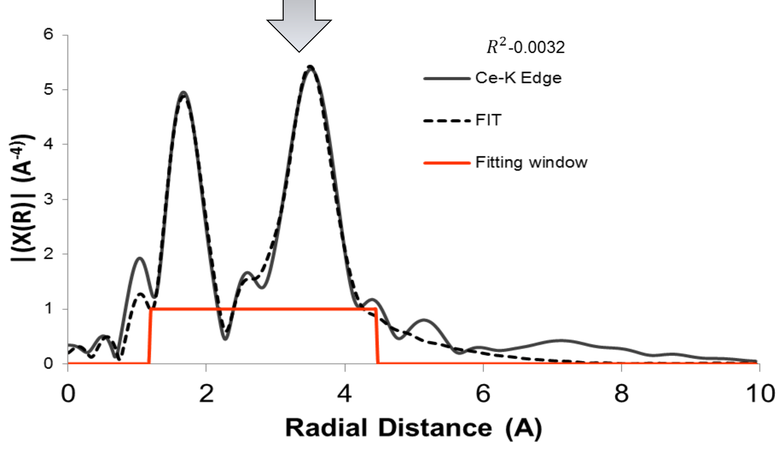
- •No diffraction patterns from iron oxides or metallic Fe were observed during reduction or reoxidation cycles.
- •Only CeO_2 reduction could be deduced by the shift in peak positions
- •Information about Fe could only be obtained through XAS

Information about Fe !!!

Ce-K edge XAS



EXAFS Structural modelling 10% Fe_2O_3-CeO_2



- **Changes in the coordination of Ce for 10% Fe_2O_3-CeO_2**
- **Model the data with factor x defined for iron incorporation: x = ~0.2**
- **Solid solution: $Ce_{0.8}Fe_{0.2}O_{1.8}$**

Conclusions

- **The in situ XRD study revealed**
  - **Stable crystallite sizes for 80% Fe_2O_3-CeO_2**
  - **Formation of solid solution in 10% Fe_2O_3-CeO_2 and fine dispersion of nano sized Fe**
- **The XAS structural modelling of 10% Fe_2O_3-CeO_2 provided information about the Ce neighbors and the modelling results suggest that 20% of the Fe is incorporated into CeO_2 forming solid solution.**

Acknowledgements

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References

[1] Galvita, V.V., et al., *CeO_2-Modified Fe_2O_3 for CO_2 Utilization via Chemical Looping.* Industrial & Engineering Chemistry Research, 2013. **52**(25): p. 8416-8426.

[2] Galvita, V., et al., *Deactivation of modified iron oxide materials in the cyclic water gas shift process for CO-free hydrogen production.* Industrial & Engineering Chemistry Research, 2008. **47**(2): p. 303-310.

[3] Adanez, J., et al., *Progress in Chemical-Looping Combustion and Reforming technologies.* Progress in Energy and Combustion Science, 2012. **38**(2): p. 215-282