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Low Temperature Reactivity of Aluminum Nanopowders with Liquid Water

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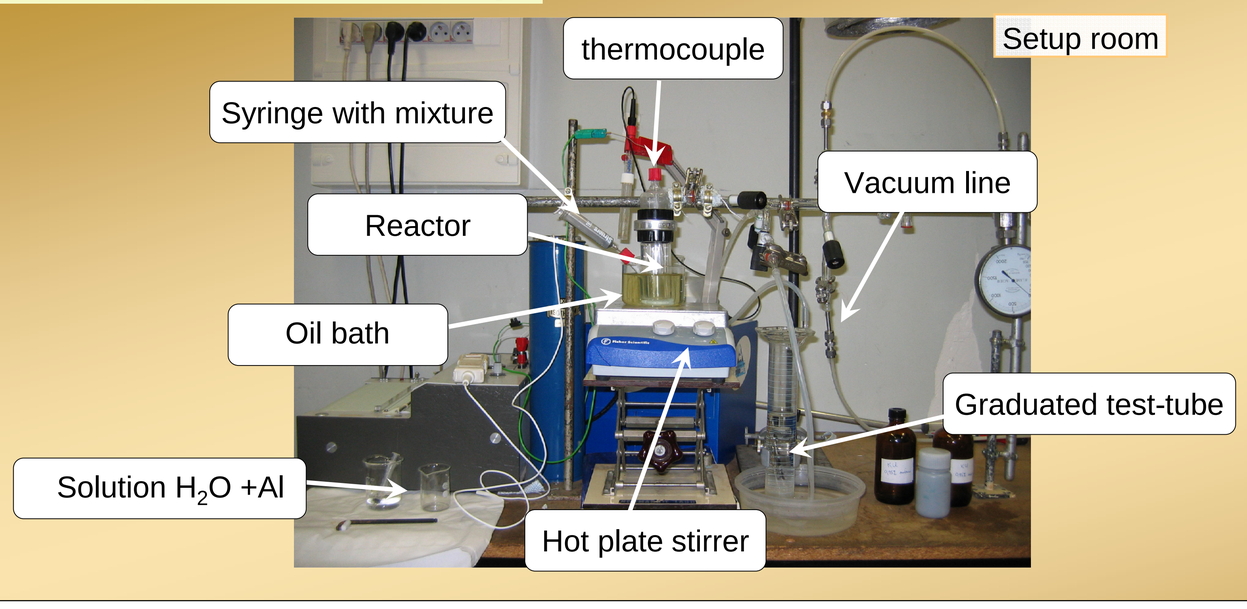
1 - Introduction

- Aluminum nanoparticles react with water and produces hydrogen.
- As hydrogen is envisaged as an alternative energy vector, is it possible to consider Ultrafine Aluminum Powder (UFP) as a in situ hydrogen source ?

2 - Objectives of the present work

- Understanding of the macrokinetics of the hydroxidation of aluminum.
- Measurements of the H_2 gas release rate as a function of time and temperature.
- Investigating the effect of salt water compared to pure water.

3 - Experimental setup



Operating Conditions

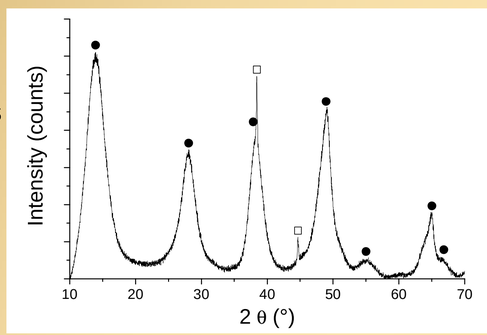
- Isothermal conditions during experiment
- Temperature parameter range [45 – 90 °C]
- Alex powder, air passivated (100 nm mean diameter)
- Mixture composition fixed as:
  - 60 g distilled water
  - 0.1 g of Alex powder
- Saline solution 3% w. (KCl or NaCl)

Experimental Techniques

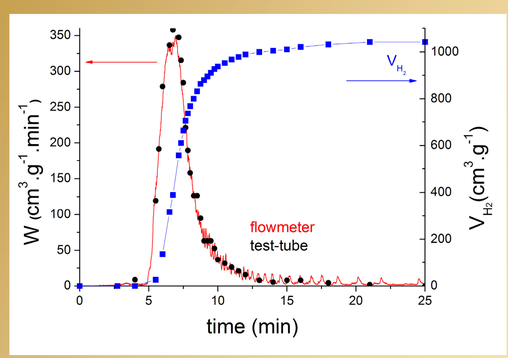
- Temperature ➪ Thermocouple
- H_2 gas release ➪ { graduated test-tube / Mass flowmeter }
- Transmission electron microscopy
- X-ray diffraction

4 - Results

- X-ray analyses of the solid reaction residues reveal aluminum oxyhydroxide AlOOH (boehmite)
- Gas-phase chromatography confirms the production of gas dihydrogen (H_2)
- $Al(s) + 2\ H_2O(l) \rightarrow AlO(OH)(s) + 3/2\ H_2(g)$



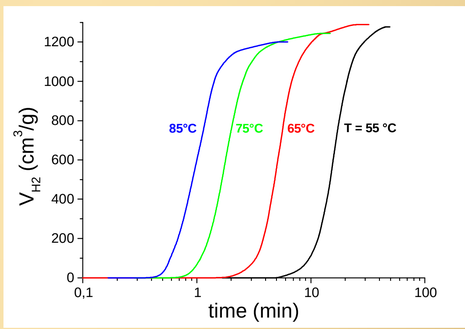
Diffraction diagram of the Al+H_2O reaction residues.
●: AlOOH (JCPDS 00-049-0133);
□: Al (JCPDS 01-085-1327)



Example of an experimental result at T=72°C

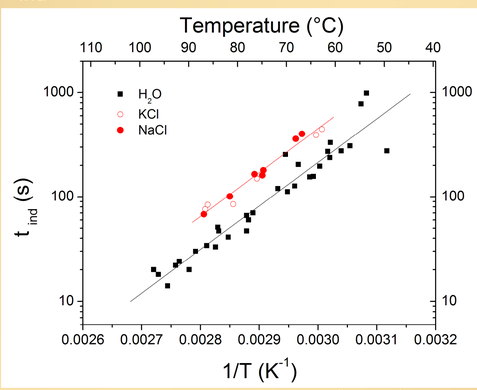
- Reaction starts after an induction period (t_{ind})
- After this period, instantaneous H_2 flow rate increases rapidly, exhibits a maximum then decreases.
- S-shaped gas evolution for the total volume of hydrogen observed.

- Temperature dependence of the induction period.
- Rat'ko et al [2] suggest that this induction period corresponds to the hydration of the natural oxide film (Al_2O_3).
- The theoretical value of maximum gas release (1350 cm³ H_2/g Al) is not reached, but conversion is quasi-complete.



Gas evolution process in water for different temperatures

- Induction time and maximal flow rate follow an Arrehnius law.
- t_{ind} for saline solutions are higher than for pure water, with lower W_{max}.

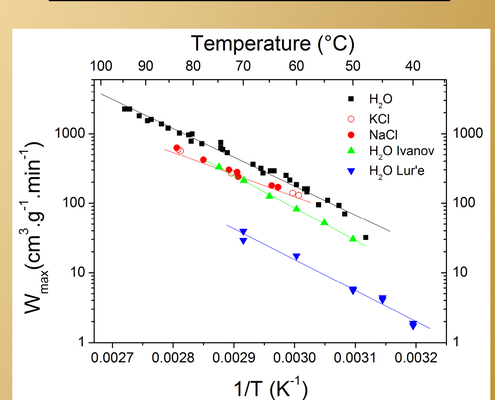


$$t_{ind} = t_{ind}^{\infty} \cdot \exp\left(\frac{-Ea}{RT}\right)$$

Solution	t_{ind}^{∞} (s)	Ea (kJ/mol)
H_2O	$6.41\ 10^{-11}$	-79.9 ± 4.1
NaCl, KCl solutions	$1.12\ 10^{-10}$	-80.4 ± 4.3

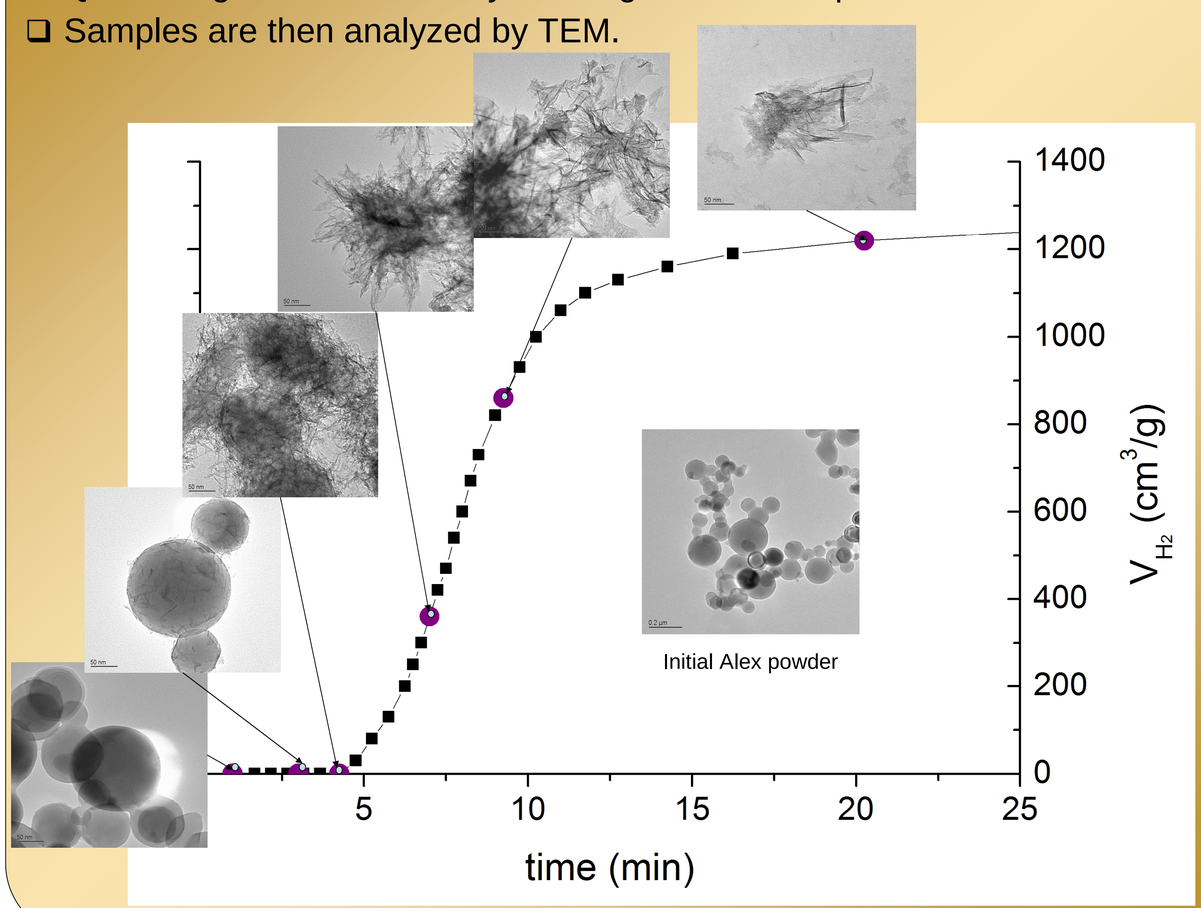
$$W_{max} = W_{max}^{\infty} \cdot \exp\left(\frac{-Ea}{RT}\right)$$

Solution	W_{max}^{∞} (cm³/g/min)	Ea (kJ/mol)
H_2O, present work	$1.90\ 10^{14}$	77 ± 1.5
H_2O, Ivanov *et al.*[1]	$7.93\ 10^{15}$	89 ± 1.5
H_2O, Lur'e *et al.* [3]	$3.02\ 10^{14}$	85 ± 4.3
NaCl, KCl solutions, present work	$7.70\ 10^{11}$	62 ± 3.5



5 - TEM Analysis

- Quenching of the reaction by freezing mixture samples at different times.
- Samples are then analyzed by TEM.



6 - Conclusions

- The temperature dependence and the influence of salinity have been investigated on the low temperature reactivity of aluminum nanoparticles with water .
- The production of hydrogen is preceded by an induction period corresponding to the hydration-dissolution-precipitation of the oxide film initially covering the particles.
- This induction period is thermally activated (Ea= -80 kJ/mol).
- The hydrogen released by the reaction (hydroxidation) is characterized by W_{max}, which is also thermally activated with similar values of Ea found in the literature.
- The hydroxidation of the nanopowder is slowed down in the presence of chloride ions.

References

[1] V.G. Ivanov, M.N. Safronov, O.V. Gravilyuk, Combust. Explos. Shock Waves, 37 (2001) 173.
[2] E. Rat'ko, V.E. Romanenkov, E.V. Bolotnikova, Z.V. Krupen'kina, Kinetics and Catalysis, 45 (2004) 141.
[3] B.A. Lur'e, A.N. Chernyshov, N.N. Perova, B.S. Svetlov, Kinet. Katal., 17 (1976) 1453.