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# XPS study of the evolution upon cycling of the interface layers on porous graphite electrodes for Li-$O_2$ batteries

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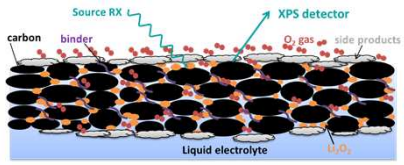
## Motivation

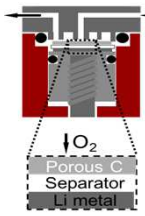
- **Li-$O_2$ batteries**: complex interface at the porous electrode
  → critical to identify the side-reaction products
- **X-Ray Photoelectron Spectroscopy** (XPS): most suitable technique to analyze the chemical composition of the extreme surface of materials (analysis depth ~ 5-10 nm)



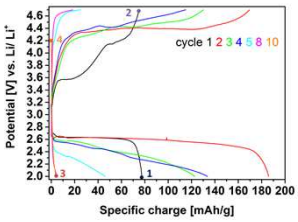

## Electrochemical data

**Electrochemical measurements**:
- porous electrode (thickness 200 µm): 85 wt % graphite SFG6 (Imerys) /
- 15 wt % PTFE
- electrolyte: 0.2 M LiTFSI in diglyme
- current density: 0.1 mA/cm²

**Sample preparation for XPS**:
- Electrodes washed in diglyme
- Samples transferred using an air-tight transfer chamber



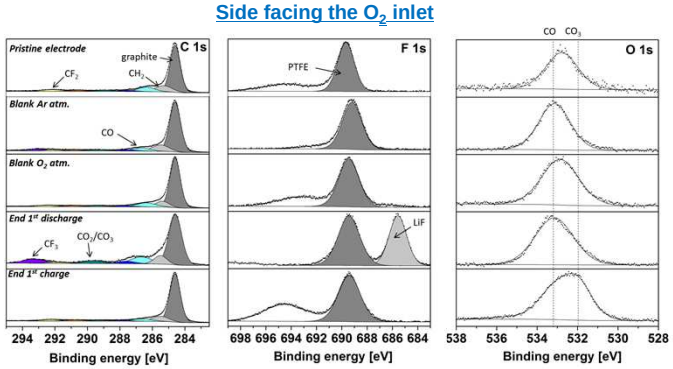

Scheme of the home-made Li-$O_2$ cell




Galvanostatic curves of the porous graphite electrode cycled vs. Li

## XPS data-1st cycle

### Side facing the $O_2$ inlet

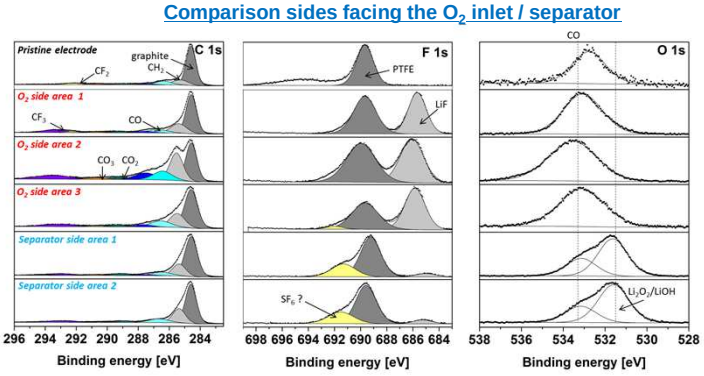



**End 1st discharge:**
- Thin interface (graphite: main component)
- Presence of LiF, $CO_3$ and CO species
- No evidence of $Li_2O$, $Li_2O_2$ or $LiO_2$
- Salt degradation

**End 1st charge:**
- Thin interface (graphite: main component)
- Spectra similar to those of the pristine electrode

→ Decomposition of the species formed upon discharge

### Comparison sides facing the $O_2$ inlet / separator



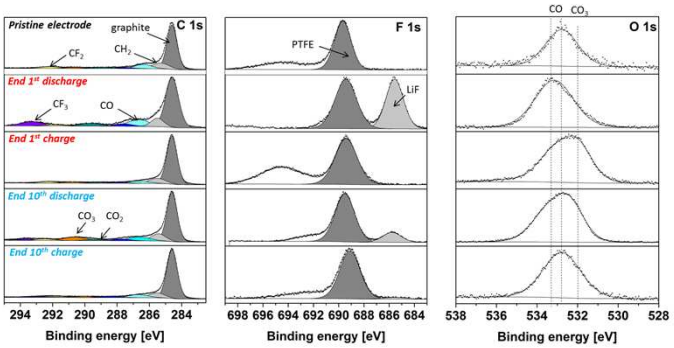

**Both sides:**
- Thin interface (graphite: main component)
- Presence of LiF, $CO_3$ and CO species

**But on the side facing the separator**
- Lower amount of LiF
- Extra contribution on the O 1s spectrum at 531.5 eV: $Li_2O_2$/LiOH* ? Ratio Li 1s/O1s (531.5 eV)~ 1

→ Inhomogeneity of the interface composition between both sides

* XPS technique not sensitive to H

## XPS data-10th cycle (side facing the $O_2$ inlet)




**End 10th discharge:**
- Thin interface (graphite: main component)
- Presence of LiF, $CO_3$ and CO species
- No evidence of $Li_2O$, $Li_2O_2$ or $LiO_2$

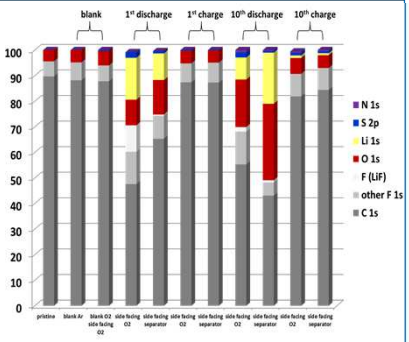
**End 10st charge:**
- Thin interface (graphite: main component)
- Spectra similar to those of the pristine electrode

→ Decomposition of the species formed upon discharge

## Summary

- ✓ No chemical reactions between the cell components during blank tests
- ✓ 1st discharge: thin interface
  - different interface layers formed on both sides of the graphite electrode
  - little amount of $Li_2O_2$ / LiOH
  - side reaction products
- ✓ Upon charge: decomposition of the products formed upon previous discharge

→ Graphitic carbon not the most suitable material
Similar study on amorphous carbon in progress




Relative contribution (at. %) of each element to the global signal for the pristine electrode and electrodes recovered after 1 and 10 cycles

## Acknowledgement

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