

Nanoscale characterization of Co and Co-B catalytic coatings before and after catalytic tests for the sodium borohydride hydrolysis

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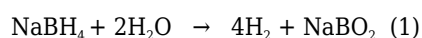
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The use of hydrogen as a potential future energy carrier is limited due to the problems of its storage. Hydrolysis of hydrogen storage materials such as sodium borohydride (NaBH₄, SB) has been one of the most investigated approaches for hydrogen generation. SB is stable in dry air and combines lightweight with high hydrogen content (10.8 wt%).



Although spontaneous, the SB hydrolysis (reaction 1) needs catalysts to occur at appreciable rates. Co has demonstrated to be a good choice because its compromise between activity and cost. However, its major drawback is related to stability: these materials deactivate upon cycling. Despite the great number of works reporting Co and Co-B based catalysts, the nature of the active phase and deactivation mechanisms are still under intense discussion.

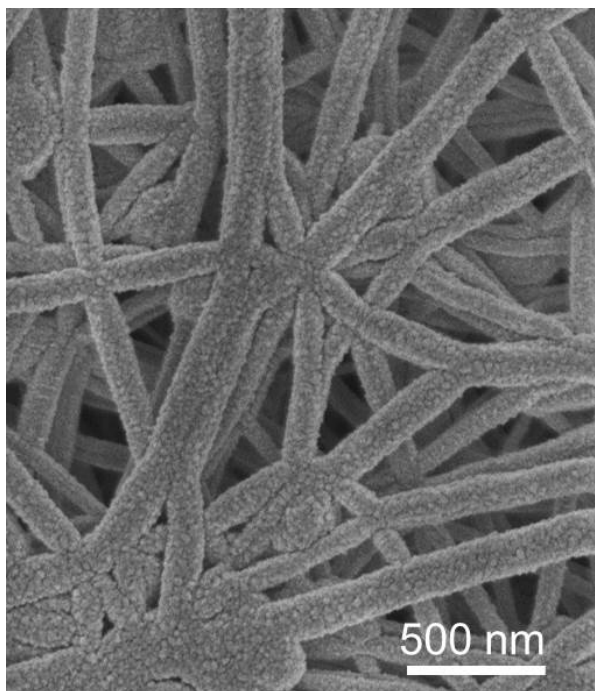
We have recently reported the preparation of supported Co metallic catalysts as thin films by magnetron sputtering for sodium borohydride hydrolysis [1]. Magnetron sputtering is a very versatile technique used in this work to fabricate Co and CoB catalytic coatings under different deposition conditions and supported on different substrates (i.e. silicon and polymeric membranes). In this work we have been able to study by electron microscopy the catalytic coatings as grown on the wires of the polymeric membranes. The structural and compositional characterization by SEM and (S)TEM techniques has been performed before and after the catalytic tests (19 wt% SB in NaOH 4 wt%, 90 min reaction time). Fig. 1 and 2 show the SEM morphology of a Co thin film on the polymeric membrane before and after the catalytic test, respectively, showing the growth of a new layer onto the catalysts upon operation. Further nano-analysis of the structure and compositional distribution have been performed by (S)TEM techniques coupled to EELS. They also reveal the formation of fiber/nanoflake-like nanostructures onto the catalytic coatings (Fig. 3 and Fig. 4). Compositional analysis have pointed out the formation of Co-borates and most likely cobalt oxide/hydroxide nanoflakes (i.e. CoO(OH)) which could be the origin of the leaching and deactivation mechanisms of the Co-based catalysts for the investigated reaction.

Acknowledgements:

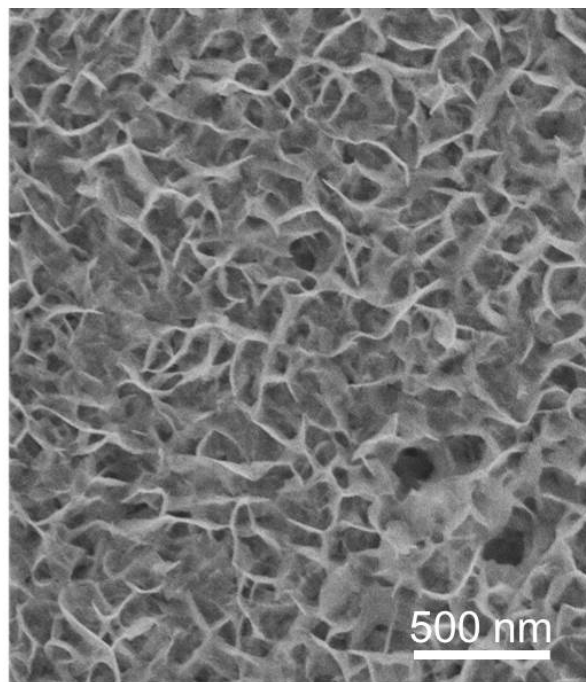
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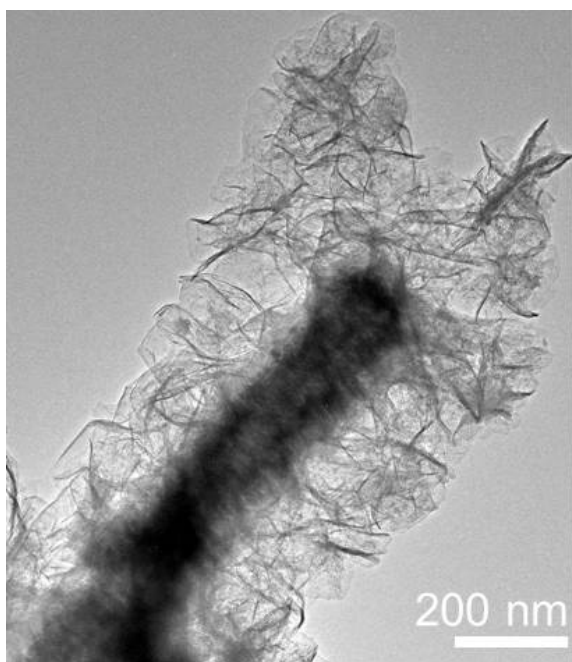
[1] Paladini, M. et al. Applied Catalysis B-Environmental 2014, 158, 400-409.



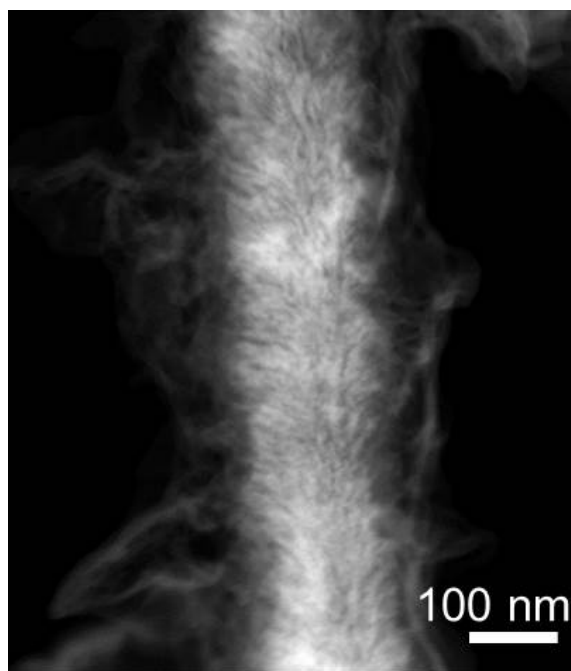
SEM image of a Co-thin film on polymeric membrane before the catalytic test.



SEM image of a Co-thin film on polymeric membrane after the catalytic test



TEM image of a Co thin film supported on polymeric membrane after the catalytic test.



STEM image of a Co thin film supported on polymeric membrane after the catalytic test.