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FIB and TEM Study of Reactivity of Co Doped $BaCe_{1-X}Zr_XO_3$ Membrane in Reducing Environment

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Continuously diminishing fossil fuel resources and environmental concern provides an increasing motivation to search for alternative energy sources. Out of several possible resources, hydrogen produced by catalytic reforming of hydrocarbons has the potential of commercial exploitation and meeting part of this Nation's energy needs. Unfortunately, catalytic reforming always accompanies the generation of other by-products, which has deleterious effects on the materials and on the environment. Higher operational temperatures that are needed in order to achieve sufficient conductivity add to the problem of materials degradation.

It is a major challenge to develop new materials that will catalytically reform hydrocarbons at relatively lower temperature and separate out hydrogen from the reaction product mixture. Perovskite-based materials are good candidates for this type of multifunctional operation. Addition of zirconium adds to the stability and doping with Co may add to the conductivity of the material [1-2].

The homogeneous single-phase material was synthesized using the oxalate co-precipitation technique followed by heat treatment at 1550 ^{0}C for 12 hours [3]. The material was compacted and sintered at 1550 ^{0}C to produce the membrane and then reduced at 600 and 927 ^{0}C in a 10% wet-H_2 environment for 24 hours. Before and after the reduction, the membrane was FIB sectioned with a FEI Strata 400s Dual Beam FIB and characterized by JEOL 2010 FasTEM. Analytical imaging of the material was carried out with a Tecnai T12 TEM equipped with STEM-EDS.

Microscopy observation indicates that the as-synthesized membrane is totally crystalline with polygonal grains. In localized grain-boundary regions exsolution of Co in the form of CoO can be observed (Figure 1). After heating to 600 ^{0}C in a reducing environment, extensive chemical changes take place in the grain bodies and boundaries. After this reduction the material is no longer homogeneous, instead a large number of pores and cracks could be seen in the material. After reduction at 927 ^{0}C the extent of degradation is even more pronounced. Single-phase barium cerate zirconate is still present; BaO, CeO_2 and $BaZrO_3$ phases are located in the grain-boundary region (Figure 2). The material appears to have a strong tendency to absorb hydroxyl ions and hydrolyze.

Materials chemistry clearly plays a significant role in determining the performance of the material in fuel cell. Lack of thermodynamic data, phase diagram information poses limitation in material selection and design. Research vary the level of doping in this material to synthesize barium cerate zirconate with optimum properties and better stability will be described in this presentation.

References

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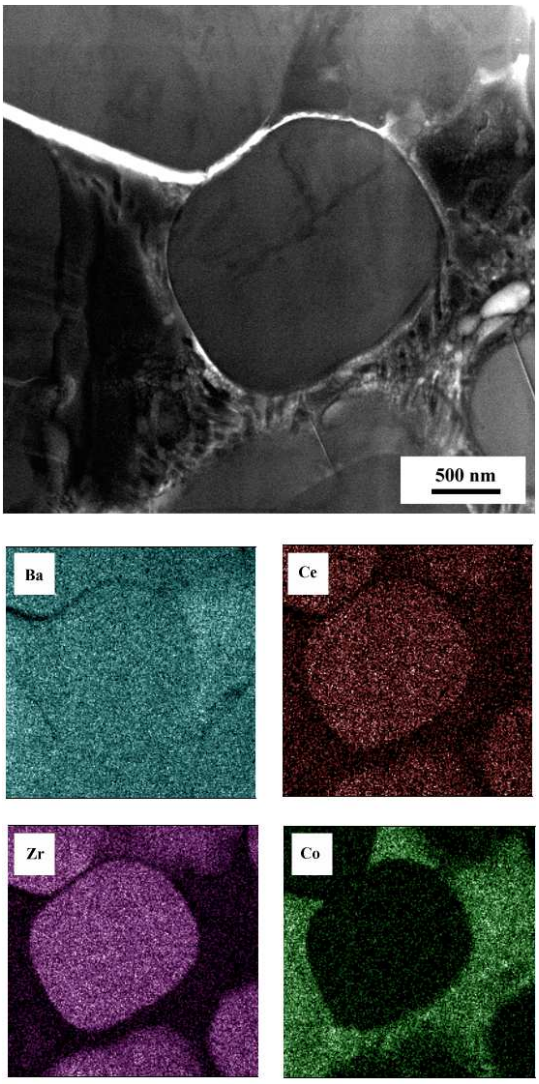

Figure 1: BF image and STEM-EDS maps of a cross-section of as-synthesized Co-doped $BaCe_{1-X}Zr_XO_3$ membrane. Localized exsolution of Co takes place to produce a Co-rich phase at the grain boundaries.

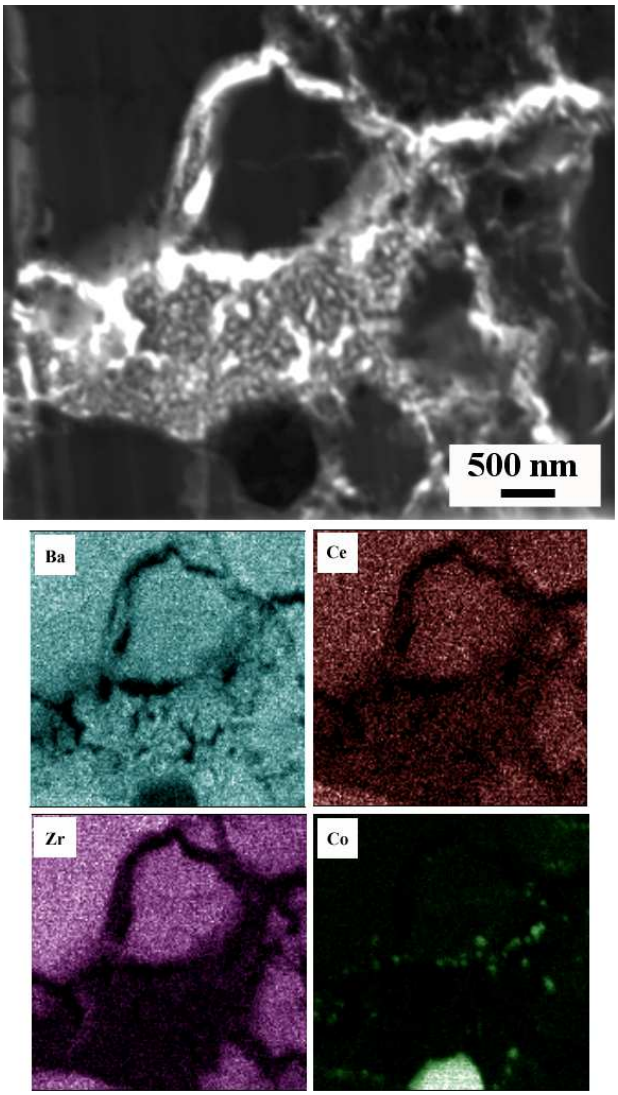

Figure 2: BF image and STEM-EDS maps of a cross-section of the Co-doped $BaCe_{1-X}Zr_XO_3$ membrane after reduction at 927 ^{0}C for 24 hours. Separation of elements by hydrolysis can be observed. The degradation of the intergranular regions is clearly visible in the BF image.

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