Microwave assisted hydrothermal synthesis of NiO-$Ce_{1-x}Eu_xO_{2-y}$ powders for fuel cell catalytic anodes

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CeO_2-doped with rare earth ions (RE^{+3}) can be used as an alternative material to traditional YSZ anodes, due to higher activities and lower ohmic losses in intermediate-temperature SOFCs (500-800°C). The present study reports the synthesis of NiO-$Ce_{1-x}Eu_xO_{2-y}$ powders using the microwave-assisted technique and the resulting powder characteristics that benefit their use as SOFC anode material.

All NiO-$Ce_{1-x}Eu_xO_{2-y}$ where x = 0.1 (H1); 0.2 (H2) and 0.3 (H3) compositions had 50% of NiO to produce ~39% metallic Ni after reduction, which is considered ideal for SOFC anode applications [1]. The microwave assisted hydrothermal synthesis is divided into two steps. In the first one, an ion cerium solution was prepared adding deionized water. The solution was agitated and heated up to 80°C until the total dissolution of cerium nitrate. Then, Europe nitrate was added, followed by ammonium hydroxide solution. The final solution had its pH adjusted. The hydrothermal synthesis took place at 140°C for 30 minutes at a heating rate of $10°C/min^{-1}$. The solution was then cooled to ambient temperature and the precipitate removed. In the second step, the precipitate was dissolved. Then, Nickel nitrate in ammonium hydroxide solution was prepared and added to the precipitate. The pH was adjusted and the new solution was submitted to the same hydrothermal treatment for 1 hour, resulting in the final powder, that was washed and dried at 60ºC during 24 h. The powders were calcined and characterized by XRD, TG, TPR and SEM-FEG.

The TPR profiles of the powders are shown in Figure 1. All materials depicted two reduction events. The first event took place between 345 and 450°C and was probably due to the first reduction of NiO. Two peaks can be seen ~ 370 ºC indicating that the reduction of NiO was followed either by the reduction of cerium ions or by the interaction of cerium ions to stabilize Ni during the reduction process. The second event took place between 500 and 600 ºC and corresponded to the second partial reduction of NiO.

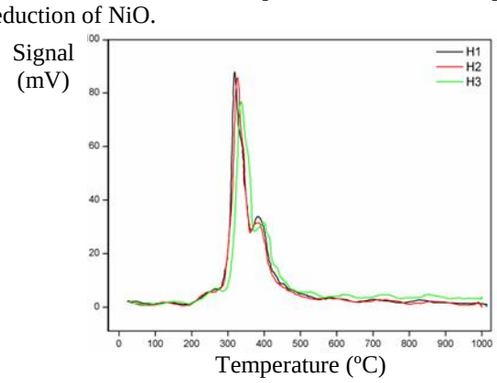

Figure 1: TPR profiles of NiO-Ce1-xEuxO2-y powders.

The XRD patterns of the obtained powders are shown in Figure 2. All materials consisted of a mixture of $Ce_{1-x}Eu_xO_{2-y}$ solid solution with cubic fluorite phase (Fm3m spacial group) and NiO. Rietveld refinement of the obtained powders showed small crystallite sizes ranging from ~13 nm to ~23 nm and superior composition control. These characteristics, along with homogeneous distribution of NiO particles are consequences of the direct and homogeneous heating and synthesis conditions of the microwave assisted synthesis. The association of the hydrothermal process with microwave radiation reduces the thermal gradient and contributes to homogeneous nucleation, uniform particle growth and, consequently, a uniform distribution of particle sizes. All these aspects are positive for SOFC anode applications. A typical SEM-FEG image of these powders is shown in Figure 3.

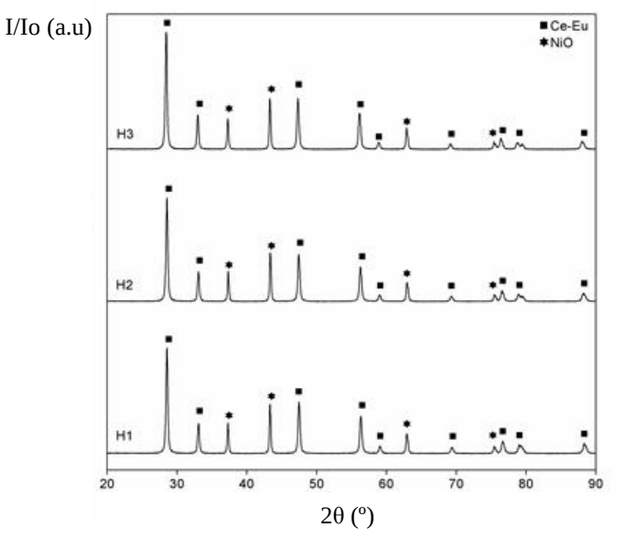

Figure 2: XRD profiles of NiO-$Ce_{1-x}Eu_xO_{2-y}$ powders.

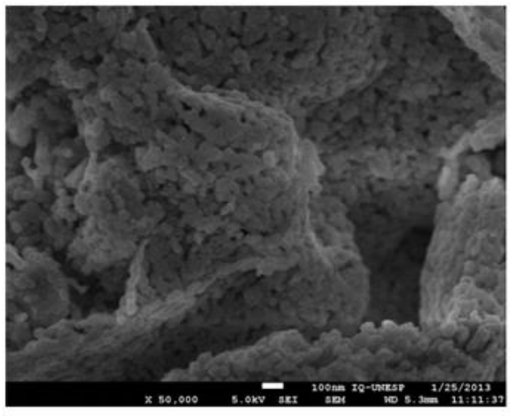

Figure 3: SEM-FEG image of H2 powder (NiO – $Ce_{0.8}Eu_{0.2}O_{1.95}$).

Acknowledgements

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References

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