



## XRD and TPR Characterization of Fe-Ni/YSZ-GDC for Application as SOFC Anode

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**Abstract** – Ni-Fe/YSZ-GDC were synthesized by Pechini method and analyzed by XRD and TPR. Results showed Ni and Fe formed a mixed oxide of spinel structure. After reduction in hydrogen presence, Fe and Ni formed a solid solution. TPR results showed that there were nickel and iron species with different reduction temperatures. The species reduced at lower temperature were related to smaller crystallites of nickel, iron or NiFe oxide and those at higher temperatures were attributed to nickel or iron species with strong interaction with oxygen vacancies of the support.

In recent years, solid oxide fuel cells (SOFCs) have received considerable attention as promising devices for the future energy economy. Mostly due to some of their unique advantages, such as high efficiency, low pollutant emission, fuel flexibility and possibility of use in remote places (1-4). Ni-YSZ (nickel supported on zirconia stabilized with yttria), with a mass ratio of 35% Ni and 65% of YSZ, is commonly used as anode in SOFC, due to its excellent electrochemical performance when using hydrogen fuel. However, nickel is susceptible to strong loss of catalytic activity by carbon deposits or sulfur deactivation, harmful to the anode performance, especially when the cell is fed with oil or alcohol (5). To take advantage of nickel use as catalysts in SOFC anodes, several groups have studied the addition of other metals in order to minimize the problem of high carbon deposition (6). This work aim at producing electrocatalysts based on nickel and iron supported on YSZ and GDC by Pechini method, with metal content of 35% by weight.

The samples were prepared by Pechini method (6, 7). All samples were prepared to contain 35% of metal on the support. The support was a mixture of 70% of YSZ [(Y<sub>2</sub>O<sub>3</sub>)<sub>0.08</sub> (ZrO<sub>2</sub>)<sub>0.92</sub>] and 30% of GDC (Ce<sub>0.9</sub> Gd<sub>0.1</sub>O<sub>2</sub>), mixed in ball mill for 24 hours. Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and Fe (NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O were the precursor for nickel and iron, respectively. For comparison purposes, a sample was prepared containing only nickel (35%), prepared by the mechanical mixing of NiO with the support in a ball mill for 24 hours. All samples were calcined for three hours at 900°C in air.

The samples were characterized by X-ray diffraction (XRD) with an Shimadzu apparatus, XRD 6000, with CuK $\alpha$  radiation (40 kV and 40mA), between 10 and 80° at a rate of 0.25° per minute. The XRD analysis showed that the nickel and iron oxides were in cubic structure on the support in the calcined monometallic samples. Nickel and iron bimetallic samples were associated in the form of mixed oxide (NiFe<sub>2</sub>O<sub>4</sub>), spinel type. Temperature programmed reductions (TPR) of the same samples were conducted from 30 to 1100°C at a rate to 10° per minute under 1.5% mol H<sub>2</sub>/air at a flow rate of 0.5 ml s<sup>-1</sup>. The monometallic catalyst samples prepared by Pechini method were completely reduced below 1000°C, while for the NiO mechanical mixed sample, only 14% molar was reduced. The reduction of the bimetallic samples varied between 88 and 98% molar, increasing as the nickel load decreased. This effect may be attributed to the NiFe alloy formation. Additionally there were formation iron and nickel species with different metal-support interaction. The species reduced between 400 and 500°C were related to small crystallites and the species reduced between 500 and 900°C were those with stronger interaction with the support, probably because of strong bond formation between Fe and Ni oxides and oxygen vacancies of the support surface<sup>8</sup>. The presence of several active metal species on the surface of electrocatalysts prepared by Pechini method the unique properties of these material for the ethanol stem reforming.

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