Electronic Properties Study of Ni/MgO Nanoparticles by (XPS)

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Abstract—A lot of knowledge has been accumulated on the metal clusters supported on oxide surfaces because of their multiple applications in microelectronics, heterogeneous catalysis and magnetic devices.

In this work, the surface state of Ni / MgO has been studied by XPS (X-ray Photoemission Spectroscopy). The samples were prepared by impregnation with ion exchange Ni2+ / MgO, followed by either a thermal treatment in air (T = 100 -350 °C) or a gamma irradiation (dose 100 kGy, 25 kGy dose rate h -1). The obtained samples are named after impregnation NMI, NMR after irradiation, and finally NMC(T) after calcination at the temperature T (T = 100-600 °C).

A structural study by XRD and HRTEM reveals the presence of nanoscaled Ni-Mg intermetallic phases (Mg2Ni, MgNi2 and Mg6Ni) and magnesium hydroxide.

Mg(OH)2 in nanometric range (2- 4 nm). Mg-Ni compounds are of great interest in energy fields (hydrogen storage…).

XPS spectra show two Ni2p peaks at energies of about 856.1 and 861.9 eV, indicating that the nickel is primarily in an oxidized state on the surface. The shift of the main peak relative to the pure NiO (856.1 eV) suggests that in addition to oxygen, nickel is engaged in another link with magnesium. This is in agreement with the XPS spectra which present an overlap of peaks corresponds to NiO and MgO, at a calcination temperature T ≤ 300 ° C.

Keywords— nanoparticles, Ni-MgO, XPS, XRD.

I. INTRODUCTION

The nanoparticles usually possess unusual electronic, structural, magnetic, chemical properties [1, 2, 3], and these make them very different from the bulk materials due to their extremely small particle size (in the same range as the characteristic sizes of the physical properties). Particles in nanometric scale are of great interest in the energy field. As first characteristic sizes of the physical properties). Particles in nanometric scale are of great interest in the energy field. As first example in heterogeneous catalysts, they have a large specific surface area, when the size decreases, the surface-volume ratio increases and it provides a large contact surface. A high surface area encourages meetings with the reactants, from which a large number of reaction sites. So, the nanosized particles, monodispersed and stable, have good catalytic efficiency [4, 5].

As second example in hydrogen storage Mg-based materials are promising [6, 7], they have a relatively high hydrogen capacity [8] and higher stability in air spatially Ni-Mg nanoparticles with their low weight and low cost.

The produced nanoparticles can be characterized with several techniques: DRX and TEM for structural study, SEM for morphological one, when XPS for surface study. This last attracted wide interest among specialists in catalysis, they need know all about surface state (Catalysis is by definition a surface phenomenon). This technique is very useful because of its sensitivity to the top first layers of solid materials, and it focuses on chemical state of all elements, namely the oxidation state and covalency or ionic types of bondings [9].

The aim of this work is to study Ni/MgO materials. After nickel ion adsorption on magnesium oxide, the samples are calcinated or radiated at several temperatures. XRD, TEM and XPS study their structural properties, whereas XPS study their surface properties. This last is used to compositionally and structurally characterize of the Ni-MgO surface after impregnation step, then after exposure to gamma irradiation and after calcination at several temperatures.

The aim of this work is the study of electronic properties of Ni/MgO composites basing essentially on surface study by XPS technique.

II. EXPERIMENTAL

A. The characterization of samples was done by Emperean Panalytical (XRD diffractometer apparatus). XPS characterization was carried by using K-alpha surface analysis of THERMO-SCIENTIFIC fully automated. The compositions and chemical states of the constituents for the samples were analyzed using a monochromatized Al Kα line

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(1486.5 eV) in the Laboratory of Law temperatures and nanostructures, Alicante.

III. RESULTS AND DISCUSSION

The X-ray diffraction spectra of the MgO, Ni2+/MgO support after impregnation and that irradiated one are reported in the figure 1. The X-ray diffraction of the support (MgO) shows characteristic peaks of the magnesia (periclase) in the CFC structure with Fm3m symmetry. After adsorption of nickel (impregnation stage), the NMI and NMR spectra exhibit spikes corresponding to magnesium hydroxide as well as Ni-Mg intermetallic compounds (Mg2Ni and MgNi2 with hexagonal structure and Mg6Ni with cubic structure). There is an overlap of the peaks, and it was difficult to identify them because of the same structure and the atomic rays of Ni and Mg that are very close.

Fig. 2 shows the X-ray diffraction of the impregnated and calcined samples at several temperatures: MNC (T = 150, 300, 400, 500, 600, 700 °C). At low temperature, the peak at 38 ° and characteristic of Mg(OH)2 plus Mg6Ni while that at 58,60 ° may be due to the presence of Mg(OH)2 and MgNi2, when the temperature increases more than 300°C, Mg(OH)2 disappears under the effect of thermal decomposition into magnesium oxide at 310°C [12]. At temperature T > 300 °C, the peaks become narrower and the majority of the peaks corresponding to the intermetallic phases disappear and all those of the magnesium oxide probably appear these compounds are not well crystallized. The interaction between magnesium and oxygen becomes stronger compared to the interaction between magnesium and nickel.

The XPS technique allows to know the chemical nature (state of oxidation) of the different constituents of the samples, as well as their surface state. X-ray photoelectron spectroscopy was carried out and showed a change in the position of the Ni and Mg peaks with respect to the mass state.

The XPS spectra are shown in Fig. 3. These results show that in the case of irradiated, impregnated and heat-treated (calcination) samples, nickel is essentially in the form of Ni2+ on the surface of all our samples (NMR, NMI and NM (T) (T = 100, 300 and 350 °C).

Ni2p has two peaks at energies of the order of 856.1 and 861.9 eV, indicating that nickel is mainly oxidized at the surface [13]. The shift of the main peak from that of pure NiO (856.1 instead of 854.0 eV) suggests that in addition to oxygen, nickel is involved in another bond with magnesium (The higher binding energy implies that Ni2+ does not exist in the free NiO form and a strong interaction between the Ni2+ and MgO species [14].

This is in agreement with the oxygen spectra which have an overlap of the O1s peaks corresponds to NiO and MgO [15], for a calcination temperature T ≤ 300 °C.
IV. CONCLUSION

After adsorption of nickel on MgO (impregnation stage), Ni-Mg intermetallic compounds (Mg2Ni, Mg6Ni and Ni2Mg) are detected by X-ray diffraction, and confirmed by the XPS technique, at the surface, the presence of a bond between nickel and magnesium in the second neighborhood causes a shift of the nickel oxide energy level.

REFERENCES