Surface segregation effect in nanocrystalline Mg-Ni alloys and composites

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Polycrystalline Mg-Ni alloy thin films were prepared onto glass substrates using computer-controlled ultra high vacuum (UHV) magnetron co-sputtering. The chemical composition and the cleanness of all layers was checked *in-situ*, immediately after deposition, transferring the samples to an UHV (4×10^{-11} mbar) analysis chamber equipped with X-ray photoelectron spectroscopy (XPS). The thickness and composition of the deposited films were determined using X-ray fluorescence analysis (XRF).

Nanocrystalline Mg_2Ni - type alloys were prepared by mechanical alloying (MA) followed by annealing. The MA process has been studied by X-ray diffraction (XRD), scanning electron microscopy and atomic force microscopy (AFM). Formation of the nanocrystalline alloys was achieved by annealing of the amorphous material in high purity argon atmosphere at 723 K for 0.5 h. The average size of nanocrystalline grains, according to AFM studies, was of the order of 30 nm. Furthermore, we have also prepared Mg_2Ni/Pd composites with Pd content up to 10 at. %.

The experimental XPS valence bands measured for MA Mg_2Ni nanocrystalline alloys and Mg_2Ni/Pd composites showed a significant broadening compared to those obtained for polycrystalline Mg-Ni alloy thin films. This is probably due to a strong deformation of the nanocrystals in the MA samples. The strong modifications of the electronic structure of the nanocrystalline Mg_2Ni-type alloys as well as Mg_2Ni/Pd composites could significantly influence on their hydrogenation properties, similarly to the behaviour observed earlier for the nanocrystalline FeTi- and $LaNi_5$-type alloys.

The bulk and surface chemical compositions of the samples were measured by XRF and XPS, respectively. Results on XRF measurements revealed the assumed bulk chemical composition of the polycrystalline and nanocrystalline samples. On the other hand, core – level XPS showed that the surface segregation of Mg atoms in the MA nanocrystalline samples is stronger compared to that of polycrystalline thin films. Especially, a strong surface segregation of Mg atoms we have observed for the Mg_2Ni/Pd composites. In that case, Ni and Pd atoms are practically absent on the composite surface. On the other hand, Mg atoms strongly segregate to the surface and form a Mg based oxide layer under atmospheric conditions. The oxidation process is depth limited such that an oxide-covering layer with a well-defined thickness is formed by which the lower lying metal is prevented from further oxidation. In this way one can obtain a self-stabilised oxide-metal structure. The lower lying Ni and Pd atoms form a metallic subsurface layer and are responsible for the observed relatively high hydrogenation rate. The surface segregation process of Mg atoms in Mg_2Ni/Pd composites is stronger compared to that observed for the Mg_2Ni nanocrystalline alloy.

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