XPS valence band studies of Mg_2Ni/Pd nanocomposites

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Magnesium-based hydrogen storage materials have been considered to be possible candidates for energy storage. In this contribution we present results on valence band studies of Mg_2Ni/Pd nanocomposites using X-ray photoelectron spectroscopy (XPS). The shapes and positions of the valence bands determined for the nanocomposites were also compared with that measured for an in-situ prepared polycrystalline Mg_2Ni thin film. The structure of the samples has been studied by X-ray diffraction (XRD) with Co-K_α radiation, scanning electron microscopy (SEM) and atomic force microscope (AFM). Their bulk chemical compositions were measured using X-ray fluorescence (XRF) method.

Mg_2Ni/Pd (I) composite was prepared by milling of nanocrystalline Mg_2Ni powder mixed with 10 wt.% Pd powder (74 μm, purity 99.95 %) for 1 h in a SPEX Mixer Mill. The weight ratio of hard steel balls to mixed powder was 30:1. The nanocrystalline Mg_2Ni powder was prepared earlier by mechanical alloying (MA) followed by annealing. The mill was run up to 90 h for every powder preparation. The as-milled amorphous powders were heat treated at 723 K for 1 h under high purity argon to form an ordered nanocrystalline phase. The average size of nanocrystalline grains, according to AFM studies, was of the order of 30 nm. Mg_2Ni/Pd (II) composite was prepared by high energy ball milling (HEBM) for 1h of amorphous Mg_2Ni mixed with 10 wt.% Pd powder. Mg_2Ni/Pd (III) composite was prepared by MA for 48 h followed by annealing at 723 K for 1 h of 2Mg and Ni powders mixed with 10 wt.% Pd powder.

XPS measurements for the "as prepared" Mg_2Ni/Pd (I) composite showed two peaks (near 3.5 and 7 eV) in the valence band. These peaks disappear after UHV annealing at 738 K for 1 h. The above behaviour could be explained mainly by non-uniform distribution of Pd in the MA nanocrystalline Mg_2Ni material. After UHV annealing, the distribution of Pd in the nanocrystalline Mg_2Ni becomes more uniform and the two peaks observed for the as-prepared sample disappear. The different distribution of palladium atoms was also revealed in the SEM studies of the Mg_2Ni/Pd (II) and (III) composites.

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_2Ni 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.

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