

## Surface segregation effect in nanocrystalline $\text{Mg}_2\text{M}$ ( $\text{M}=\text{Ni}, \text{Cu}$ ) alloy thin films and bulk materials

L. Smardz<sup>1</sup>, K. Smardz<sup>2</sup>, I. Okonska<sup>2</sup>, and M. Jurczyk<sup>2</sup>

<sup>1</sup>*Institute of Molecular Physics, Polish Academy of Sciences  
M. Smoluchowskiego 17, 60-179 Poznań, Poland*

<sup>2</sup>*Institute of Materials Science and Engineering, Poznań University of Technology  
M. Słodowska-Curie Sq. 5, 60-965 Poznań, Poland*

Nanocrystalline  $\text{Mg}_2\text{M}$  ( $\text{M}=\text{Ni}, \text{Cu}$ ) alloy thin films were prepared onto glass substrates at 283 K using 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 ( $4 \times 10^{-11}$  mbar) analysis chamber equipped with X-ray photoelectron spectroscopy (XPS). All emission spectra were measured immediately after sample transfer in a vacuum of  $8 \times 10^{-11}$  mbar. The thickness and composition of the deposited films were determined using X-ray fluorescence analysis (XRF).

Nanocrystalline Mg-based alloys were prepared by mechanical alloying (MA). The 2Mg-Ni and 2Mg-Cu powder mixtures milled for more than 40h and 18h have transformed directly to a hexagonal- and an orthorhombic-type phases, respectively. The MA process has been studied by X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The average size of nanocrystalline grains, according to AFM studies, was of the order of 30 nm.

The bulk and surface chemical compositions of the bulk nanocrystalline samples were measured by XRF and XPS, respectively. Results on XRF measurements revealed the assumed average chemical composition of the samples. On the other hand, core – level XPS showed that the surface segregation of Mg atoms in the MA bulk  $\text{Mg}_2\text{M}$  nanocrystalline samples is stronger compared to that of nanocrystalline  $\text{Mg}_2\text{M}$  thin films. This is probably due to a significantly greater surface roughness of MA materials compared to that measured for sputtered thin films. Furthermore, the MA process introduced into the material a significant amount of strain, chemical disorder and defects. Results on XPS measurements combined with successive  $\text{Ar}^+$  ion beam etching showed that Ni or Cu atoms are practically absent on the MA  $\text{Mg}_2\text{M}$  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 (Ni or Cu) is prevented from further oxidation. In this way one can obtain a self-stabilised oxide-metal structure. The lower lying Ni or Cu atoms form a metallic subsurface layer and are responsible for the observed relatively high hydrogenation rate.

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Name of the presenting author (poster session II): Lesław Smardz  
e-mail address: smardz@ifmpan.poznan.pl  
<http://www.ifmpan.poznan.pl>