INVESTIGATION OF NANOCRYSTALLINE MATERIALS BY PERTURBED ANGULAR CORRELATION AND SUPPLEMENTING EXPERIMENTAL TECHNIQUES.

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The properties of nanocrystalline materials are essentially determined by the grain size and the properties of the grain boundaries of the respective material. Using PAC for the investigation of nanocrystalline materials new information about structural properties and, in case of nanocrystalline semiconductors, electronic properties are obtained. In combination with "classical" experimental techniques those investigations yield a comprehensive picture of nanocrystalline materials as it will be demonstrated for some selected examples.

Nanocrystalline materials were prepared by either ball milling (TiAl), pulsed electro deposition (PED; Ni, NiCu), or electro deposition under oxidizing conditions (EDOC; ZnO). For the PAC investigations, the samples have to be doped with radioactive ^{111}In atoms, what was performed *ex-situ*, i.e. after preparation by diffusion, or in case of PED and EDOC, *in-situ*.

In case of nanocrystalline TiAl, the formation of different crystalline phases as a function of composition, milling time, and temperature of a succeeding annealing treatment is demonstrated. In addition, a new phase showing the introduction of N impurities is identified.

In case of nanocrystalline Ni, after *in-situ* doping with ^{111}In a slightly reduced local magnetic field near the boundaries of the Ni crystallites is observed. From the PAC data the width of a so called magnetic boundary was estimated to about 3 nm, which is significantly larger than the crystallographic boundary. In nanocrystalline Ni doped *ex-situ*, the PAC data yield new information about the structure and thermodynamic properties of the grain boundaries. The grain growth upon thermal treatment is directly connected with the migration of the grain boundaries, what was directly shown by the PAC measurements. In addition, in nanocrystalline Ni samples, pre-annealed before diffusion of the ^{111}In probe atoms, the formation of ordered grain boundary structures could be experimentally shown to occur.

The homogeneity of nanocrystalline NiCu alloys depends on different control parameters during the electrolysis process. It turned out that the PAC investigations on those alloys are significantly more sensitive on the detection of Ni precipitates than e.g. X-ray diffraction (XRD) experiments. The high sensitivity of the PAC measurements on the formation of Ni precipitates is based on the detection of the local magnetic field present in Ni precipitates.

Nanocrystalline ZnO was characterised by different techniques: absorption spectroscopy (EXAFS), photoluminescence, transmission electron microscopy (TEM), XRD, and PAC. The temperature dependent investigations yield unique information about the grain size, the so called microstrain of the crystallites, the size dependence of the band gap, and the incorporation of In atoms as donors. Based on these comprehensive investigations, preparation conditions for doping of nanocrystalline ZnO with In donors and with a particle size of about 11 nm were determined.

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