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XAS study of carbon coated Co-Fe nanoparticles

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Results of a combined magnetic, XAS, NMR and Mössbauer spectroscopy study of new magnetic nanoparticle materials for biomedical applications are presented. The samples have been obtained by arc melting of graphite electrodes filled with metallic Co, $Co_{0.2}Fe_{0.8}$, Fe or magnetite. In order to determine the local structure, the metal valence state and the magnetic properties of the synthesised materials, X-ray Absorption Spectroscopy in the XANES and EXAFS range, Nuclear Magnetic Resonance spectroscopy, Mössbauer spectroscopy as well as Vibrating Sample Magnetometry were used as characterisation techniques.

^{59}Co NMR spectra of cobalt based nanoparticles measured at room temperature, 77 K and 4.2 K are characteristic for the fcc-Co phase. Also, the Fourier transforms of the Co;K-edge EXAFS spectra reveal the dominant fcc-Co phase. The $Co_{0.2}Fe_{0.8}$ EXAFS at the Fe;K-edge and Co;K-edge show the major bcc-Fe structure.

Mössbauer spectra of Fe and Fe_3O_4 derived nanoparticles show dominant bcc-Fe and Fe_3C sextets and a singlet arising from the smallest, paramagnetic particles. Materials derived from Fe_3O_4 do not differ significantly from those obtained by melting the Fe metal electrode. They exhibit a moderate magnetic hardness due to the nanometric size of the particles and the presence of magnetically hard cementite. The Fe:K absorption edge shape and energy as well as the EXAFS chi(R) functions show that the Fe oxidation degree and local environments are similar to those of metallic iron.

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Application of interface distribution function and correlation function methods for studying the lamellar structures of polymers using small-angle X-ray scattering

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Crystallizing polymers exhibit complex morphology involving coexistence of crystalline and amorphous regions. During crystallization of polymers from quiescent melt usually thin lamellar crystals are formed. These lamellae are arranged in stacks, with layers of amorphous material being inserted between the crystalline lamellae. Because amorphous and crystalline layers in lamellar stacks exhibit different electron densities, the small-angle X-ray scattering (SAXS) method is frequently used to evaluation of parameters describing this type of superstructure. In such case the SAXS curve exhibits a maximum which angular position corresponds to an average repeating distance of the lamellar structure. This distance called *the long period* can be estimated directly from the SAXS curve using Bragg law. Detailed analysis of the distribution of SAXS intensity requires determination of the correlation function and/or the interface distribution function (IDF). These functions allowed for determination the values of the long period (L_P), the crystalline and amorphous layer thicknesses (l_C and l_A respectively) and the local volume fraction crystallinity (Φ_L).

Application of these functions requires however substraction of contribution due to electron density fluctuation and performing a number of corrections of measured intensity. The present paper shows all steps necessary in the obtaining of these functions. In the application part of the study both correlation and interface distribution functions are used to describe the structural changes in poly(ethylene oxide) during cooling at a rate 10 ºC/min. from the melt to room temperature and during subsequent heating at the same rate from room temperature to melting temperature.