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Positron annihilation in magnetite nanopowders prepared by co-precipitation method

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Nanosized iron oxide powders are materials considered with regard to its application in medical therapy called hyperthermia [1]. Magnetite nanopowders with crystallite size varying from 6.6 to 11.8 nm have been prepared by the co-precipitation method [2]. In this study a change of a crystallite size is driven mainly by varying of initial pH of water ammonia solution in which a process of magnetite precipitation runs.

Crystallographic structure and phase composition obtained samples and the size of magnetite nanoparticles were determined by XRD method using Cu K_α radiation.
Mössbauer measurements confirmed that nanosized Fe_3O_4 free-flowing powders with sizes above 10 nm are superparamagnetic at room temperature.

Positron lifetime spectroscopy (PALS) has been used to assess defectiveness of microstructure. Experimental PALS spectra were successfully resolved into three lifetime components. The third component ($\tau_3 \approx 1.6$ ns, $I_3 \approx 0.2$ %) can be assigned to a process of *pick-off* annihilation of o-Ps formed in free space between powder particles and due to small intensity is excluded from further discussion of results. Lifetime of positron annihilating from delocalized state in perfect bulk material derived from two-state trapping model [3]:

$$\tau_b = \frac{\tau_1 \cdot \tau_2}{I_2 \cdot \tau_1 + I_1 \cdot \tau_2}, \quad (1)$$

could be determined only for reference bulk magnetite (Alfa Aesar, $d_c = 44$ μm). In all Fe_3O_4 nanopowder samples positron trapping achieves saturation, as a consequence of very high defect concentration. Two types of defects were revealed in the samples with nanometric size of grains. To the first one vacancy clusters in grain insides belongs. The second category of defects is composed of larger vacancy clusters in intergrain boundaries. In a table below the PALS parameters for bulk reference sample and for sample with the crystallite size of 10 nm are shown.

Table 1. PALS parameters for nanosized Fe_3O_4 powders.

Sample	τ_1 [ps]	I_1 [%]	τ_2 [ps]	I_2 [%]	τ_b [ps]	τ_m [ps]
Bulk Fe_3O_4 magnetite large	142.5(8)	66.72(87)	289.0(2.6)	32.80(70)	173.3(8)	193.2(1)
S3, d_c=10 nm	265.8(8.5)	22.6(2.8)	424.5(3.7)	77.2(2.7)	not determ.	391.1(3)

Contrary to the PALS results in other nanocrystalline ferrites prepared as compacted powders [4] a lifetime of positron annihilation from delocalized state shortened by positron trapping in defects is not observed in annihilation spectra of nanosized Fe_3O_4 powders.

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