

EFFECT OF THE PRECURSOR LAYER ON PROPERTIES OF NANOSCOPIC POWDERS FORMED BY THERMALLY INDUCED SOLID-STATE REACTIONS

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Effect of sample layer on properties of iron(III) oxide nanoparticles prepared by isothermal decomposition of the $\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ powder has been studied in static air conditions at reaction temperature of 175 °C using ^{57}Fe Mössbauer spectroscopy, XRD, TEM, and BET surface area measurements. In-field Mössbauer spectroscopy was applied for distinguishing and quantification of amorphous and nanocrystalline ($\gamma\text{-Fe}_2\text{O}_3$, $\alpha\text{-Fe}_2\text{O}_3$) iron oxides. Independently on the layer thickness, amorphous iron(III) oxide nanoparticles (1-2 nm) are produced as the primary decomposition product. Above a critical thickness, amorphous nanoparticles crystallize to nanocrystalline superparamagnetic maghemite ($\gamma\text{-Fe}_2\text{O}_3$, 5-8 nm), which process is accompanied by an increase of the sample temperature. With increasing thickness of ferrous oxalate layer, the crystallization of primarily formed amorphous Fe_2O_3 nanoparticles starts later with longer duration. The maghemite samples prepared above a critical weight reveal contrast properties suitable for application in magnetic resonance imaging. Below a critical thickness, however, the crystallization process proceeds very slowly without any indications of an increase of sample temperature. Hematite with a large surface area and excellent catalytic properties was clearly identified as the only crystallization product. The possibility of unique monitoring of the crystallization of amorphous phase through an increase of the sample temperature is demonstrated on other solid-state reactions including thermal conversions of iron(II) acetate and Prussian Blue.