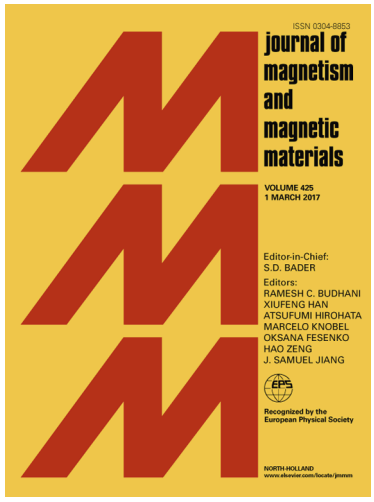


Research articles

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# Thermally induced phase transformation in multi-phase iron oxide nanoparticles on vacuum annealing

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**Abstract:** The evolution of magnetic phases in multi-phase iron oxide nanoparticles, synthesized via the transferred arc plasma induced gas phase condensation method, was investigated by X-ray diffraction, vibrating sample magnetometry and $^{57}$Fe Mössbauer spectroscopy. The particles are proposed to consist of three different iron oxide phases: α-$Fe_2O_3$, γ-$Fe_2O_3$ and $Fe_3O_4$. These nanoparticles were exposed to high temperature (~ 935 K) under vacuum ($10^{-3}$ mbar He pressure), and the thermally induced phase transformations were investigated. The Rietveld refinement of the X-ray diffraction data corroborates the least-squares fitting of the transmission Mössbauer spectra in confirming the presence of $Fe_3O_4$, γ-$Fe_2O_3$ and α-$Fe_2O_3$ phases before the thermal treatment, while only $Fe_3O_4$ and α-$Fe_2O_3$ phases exist after thermal treatment. On thermal annealing in vacuum, conversion from γ-$Fe_2O_3$ to $Fe_3O_4$ and α-$Fe_2O_3$ was observed. Interestingly, we have observed a phase transformation occurring in the temperature range ~ 498 K – 538 K, which is strikingly lower than the phase transformation temperature of γ-$Fe_2O_3$ to α-$Fe_2O_3$ (573 – 623 K) in air. Combining the results of Rietveld refinement of X-ray diffraction patterns and Mössbauer spectroscopy, we have attributed this phase transformation to the phase conversion of a metastable "defected and strained" *d*-$Fe_3O_4$ phase, present in the as-prepared sample, to the α-$Fe_2O_3$ phase. Stabilization of the phases by controlling the phase transformations during the use of different iron-oxide nanoparticles is the key factor to select them for a particular application. Our investigation provides insight into the effect of temperature and chemical nature of the environment, which are the primary factors governing the phase stability, suitability and longevity of the iron oxide nanomaterials prepared by the gas-phase condensation method for various applications.

**Keywords:** Multiphase iron oxides ($Fe_3O_4$, γ-$Fe_2O_3$ and α-$Fe_2O_3$); plasma synthesis; Mössbauer spectroscopy; X-ray diffraction; Rietveld refinement; structural and magnetic properties.

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