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Structural and Magnetic Properties of Copper-Iron Spinel / Reduced Graphene Oxide Nanocomposites

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Abstract

The effect of thermal treatment in a range of 200-500 ° C on the phase composition, morphological properties and magnetic microstructure of hydrothermal synthesized $CuFe_2O_4$ and $CuFe_2O_4$ / reduced graphene oxide was investigated using XRD, SEM, Mossbauer spectroscopy and low temperature nitrogen adsorption. The influence of reduced graphene oxide particles on the parameters of hyperfine interaction for $CuFe_2O_4$ / rGO composite was observed. The evolution of pore size distribution for synthesized samples at the increasing of annealing temperature was traced

Synthesis

Copper ferrite $CuFe_2O_4$ nanoparticles were prepared using hydrothermal route. In the first step the aqueous solution of $Fe_2(SO_4)_3$ and $CuSO_4 \cdot 5H_2O$ was prepared with 100 mL of deionized water and mixed. Then NaOH was added into the solution dropwise under vigorous stirring. The mixture was stirred for 3 hour and the prepared solution was transferred to 250 mL Teflon-coated stainless steel autoclave and heated at 150 °C for 10 h. After the cooling the obtained precipitate was separated, washed with deionized water and dried at 80 °C. The graphene oxide colloidal solution was prepared by Hummers method. [1]

Results and discussions

The broad peak on the XRD pattern of the starting powder corresponds to cubic $CuFe_2O_4$ spinel (Fig.1).The annealing in argon atmosphere causes the increasing FWHM of (311) main reflex that is an evidence of average particle size decreasing a temperature range 100-300 °C with the next stabilization and growth after annealing at 500 °C. The average particle size ferrite powder annealing in a temperature range 300-500 °C are in a range of 7-9 nm with a tendency to growing that corresponds to some decreasing of specific surface area values.

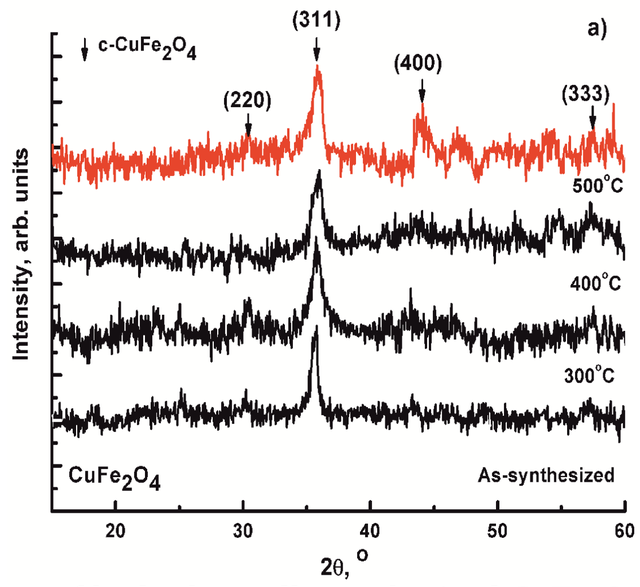

Fig.1. XRD patterns of hydrothermally synthesized $CuFe_2O_4$ samples annealed at different temperatures

The CuFe2O4 is a partially inverse spinel so cation distribution can be described as $((Cu_xFe_{1-x})_A[Cu_{1-x}Fe_{1+x}]_BO_4$, where x is the inversion parameter (x=0 and x=1 denotes the inverse and normal spinel, respectively. SEM images of as-synthesized $CuFe_2O_4$ and $CuFe_2O_4$ / rGO are presented in Fig. 2.

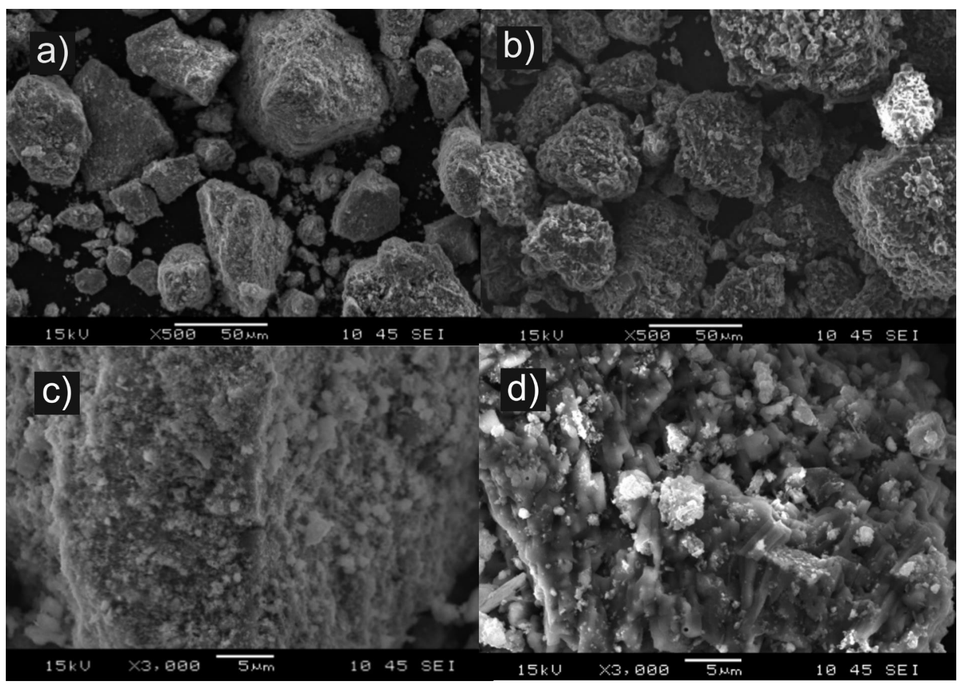

Fig. 2 SEM images of as-synthesized (a-b) $CuFe_2O_4$ and (c-d) $CuFe_2O_4$ / rGO

Room temperature (290 K) Mössbauer spectra of the as-synthesized $CuFe_2O_4$ consist of both doublet and sextet components (Fig. 3, a). The annealing causes step-by-step decrease in doublet components total intensity up to 11% for $CuFe_2O_4$ annealed at 500 ° CThe doublet components for all the cases are caused by the presence of spinel particles with sizes allowing the transition to monodomain state with magnetic moments relaxation time less compared to data measurement time (excited state lifetime of ^{57}Fe e nucleus is 141.8 ns) [2].

Mössbauer measurements were done at 90 K for verification of the superparamagnetic components` origin (Fig .3, c, d). The spectra obtained for as-synthesized $CuFe_2O_4$ consist of two ordered magnetic sextets (Fig .3, c).

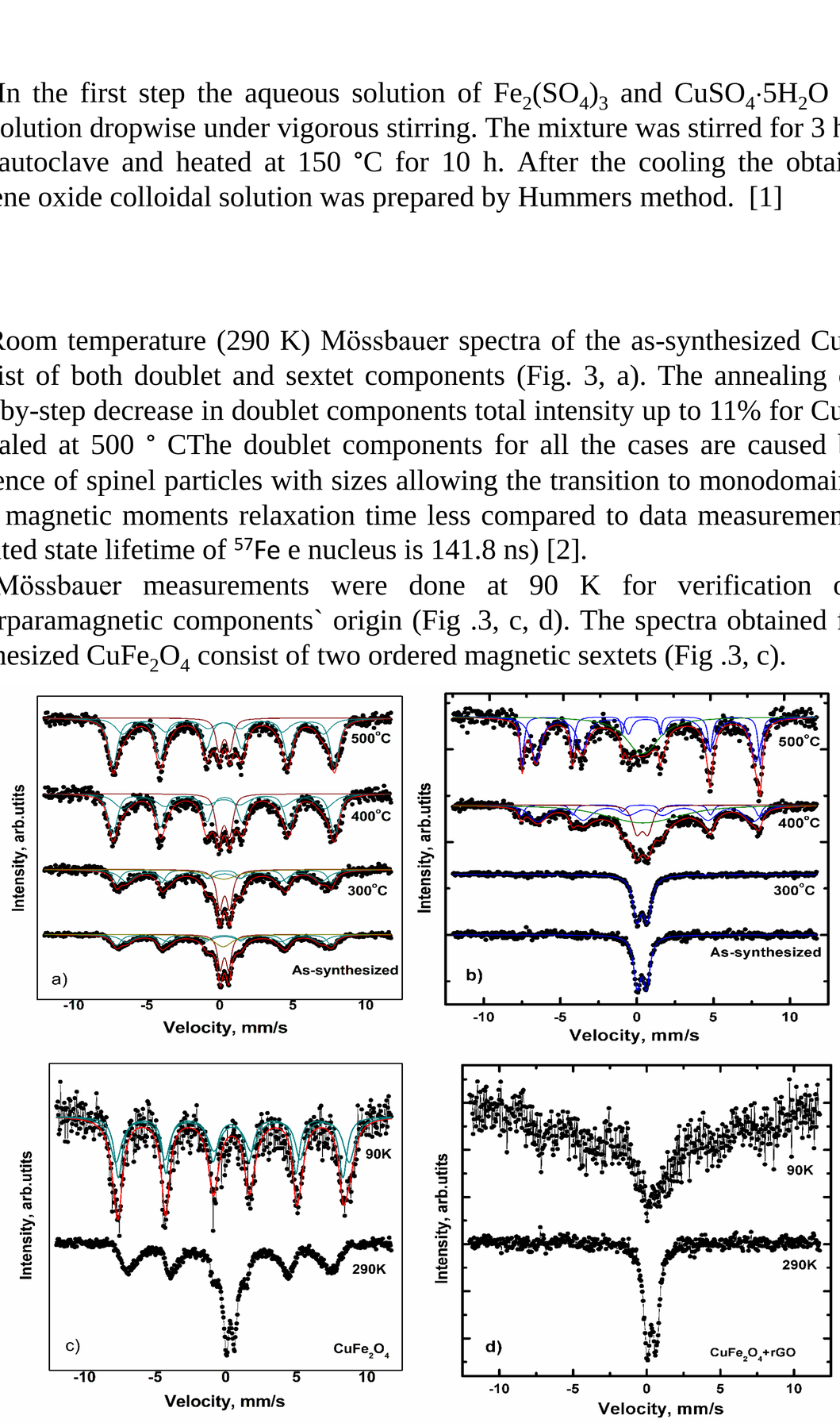

Fig.3. Mössbauer spectra of (a) $CuFe_2O_4$ and (b) $CuFe_2O_4$ / rGO annealed at different temperatures (all the spectra are measured at 290 K) and Mössbauer spectra of (c) as-synthesized $CuFe_2O_4$ and (d) $CuFe_2O_4$ / rGO measured at 290 K and 90 K

Conclusions

Ultrafine $CuFe_2O_4$ and $CuFe_2O_4$ / rGO were prepared by hydrothermal method. The effect of annealing in the range of 200-500 ° C on the phase state, morphology and magnetic properties was investigated. The experimental results showed that the crystallization of as-synthesized $CuFe_2O_4$ /rGO at annealing leads to formation both tetrahedral (t- $CuFe_2O_4$) and cubic (c-CuFe2O4) phases of copper ferrite while the materials obtained without rGO at different temperatures are ultrafine c-CuFe2O4 The inversion degree of hydrothermally obtained $CuFe_2O_4$ decrease from 1 for as-synthesized sample to 0.76 for sample annealed in argon atmosphere at 300 ° C. .

References

*1.*Boychuk, V. M., Kotsyubynsky, V. O., Bandura, K. V., Yaremiy, I. P., & Fedorchenko, S. V. (2019). Reduced Graphene Oxide Obtained by Hummers and Marcano-Tour Methods: Comparison of Electrical Properties. Journal of nanoscience and nanotechnology,19(11), 7320-7329

2. Kotsyubynsky, V., Moklyak, V., & Hrubiak, A. (2014). Synthesis and Mossbauer studies of mesoporous γ-Fe2O3. Materials Science-Poland, 32(3), 481-486