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PREPARATION AND DIFFRACTION STUDIES OF POLYCRYSTALLINE Cu-Fe MATERIALS

J. Darul *, **W. Nowicki, and P. Piszora**

Faculty of Chemistry, Adam Mickiewicz University, Grunwaldzka 6, PL-60780 Poznań, Poland

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*****) *e-mail: jola@amu.edu.pl*

Ferrimagnetic cubic spinels are technically important materials, they have been extensively investigated in order to improve good soft magnetic compounds. Considering the various spinel ferrites, cooper ferrite, $CuFe_2O_4$, has gained a prominent interest among materials science for various applications. Since of Cu^{2+} is a Jahn-Teller ion, it gives the anomalous favorable properties and also exhibits phase transition from tetragonal to cubic, depending on the temperature. $CuFe_2O_4$ can be described as a cubic close-packed arrangement of oxygen ions, with Cu^{2+} and Fe^{3+} ions at two different crystallographic sites [1-4]. The cation distribution in this oxide can be presented by the formula: $(Cu_x^{2+}Fe_{1-x}^{3+})_A[Cu_{1-x}^{2+}Fe_{1+x}^{3+}]_BO_4$. The parameter of inversion, x, is equal to 0 for inversion spinels, and to 1, when the spinel is normal. Copper ions migrate from octahedral (B-sublattice) to tetrahedral places (A-sublattice). When the spinel is synthesized using classical ceramic technologies (high temperature treatment of the initial oxides of the metal cations) with strict stoichiometry, it has a tetragonal structure of hausmannite type with crystal cell parameters a = 8.20 Å and c = 8.60 Å; c/a = 1.05. The c/a ratio can be changed via decreasing the copper concentration, or alternatively by temperature treatments [1].

A citrate process as an alternative synthesis route has been successfully employed to synthesize polycrystalline cooper iron oxide with nominal composition, $CuFe_2O_4$, with improved properties for specific applications, such as magnetic powder for massive storage devices. Corresponding amounts of the copper and iron nitrates (*Merck*) were taken in a 1:2 mole ratio along with 3 moles of citric acid (*Merck*), and dissolved in deionised water, with continuous stirring. This mixture was slowly evaporated and then dried at 120°C over night. The dried powder was crushed and calcined (300°C, 600°C and 900°C) for 5 h. The compounds formation and crystallinity of the materials were identified by XRD patterns, which were recorded on a Bruker D8 Advance diffractometer, with CuKα radiation.

Investigations on the high temperature phase transitions were carried out at the synchrotron beamline B2 at HASYLAB (DESY, Hamburg). The diffractometer was equipped with capillary furnace (STOE) and the on-site readable image-plate detector OBI. Samples mounted into quartz capillaries of diameter 0.3 mm were heated and cooled at the temperature range from RT to 900°C. The wavelength was 0.49342 Å.

The aim of the work was to establish by means of powder diffraction studies the structural properties, especially the temperature phase transition from tetragonal to cubic (400°C-425°C) and the cation distribution in $CuFe_2O_4$. Determination of the transition points, the temperature ranges of the crystalline phases coexistence, and the ions distribution in the spinel lattice, were undertaken. The structure refinement of all polymorphs using Rietveld profile analysis, based on the synchrotron X-ray data, were performed**.**

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