Synthesis of ReF_3 (Re= rare earth ions) nanoparticles for NMR research

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Studying the properties of the nanopowder DyF_3 is one of the most interesting subject in connection with the fact that DyF_3 improves the characteristics of Nd-Fe-B magnets [1-4]. The ferromagnetic phase transition in a single crystal was observed at T_c=2.55 K [5]. Investigation of Curie temperature dependence versus the size of DyF_3 nanoparticles by ^{3}He NMR is a fundamental problem. The results of the hydrothermal synthesis DyF_3 is published only in a few works [6-8] in despite of the large number of articles on the synthesis rare earths trifluorides. Therefore the synthesis of such nanoparticles is also an actual task.

In this work synthesis series of DyF_3 nanosized samples was prepared for investigation of Curie temperature dependence versus the size of DyF_3 nanoparticles by ^{3}He NMR.

The samples of DyF_3 were synthesized using following technology [9,10] with different microwave-assisted hydrothermal treatment time (0, 30, 420 min). The chemical reactions can be expressed as follows:

$$Dy^{3+} + 3F^{-} = DyF_3$$

Transmission electron microscope (TEM) images were taken with Philips CM300 operated at 300 kV (Neel Institute, Grenoble, France). Figure 1 shows the TEM image with the corresponding electron diffraction pattern in the insert and size distribution diagram for sample #3. On sharp diffraction rings can judge of the good crystallinity of samples (rings radii: 0.36 nm, 0.32 nm, 0.20 nm). The synthesized nanoparticles have average size of about 16 - 18 nm (sample #1 – 16.9 nm, sample #2 – 16.9 nm, sample #3 – 18.2 nm).

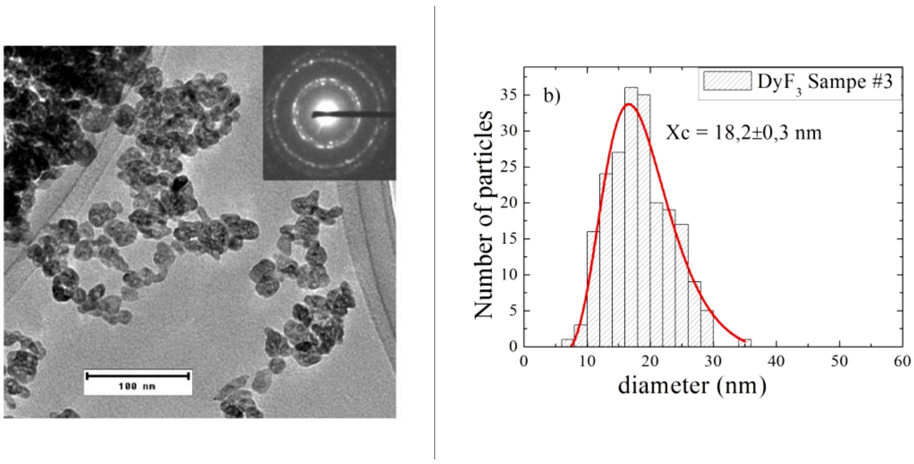

Figure 1. a) TEM image of DyF_3 nanoparticles with corresponding electron diffraction pattern in the insert (sample #3). b) The size distribution diagrams for sample #3. Solid line is the log-normal distribution fitting, X_c is the center.

Crystal structure of DyF_3 nanoparticles was characterized by X-ray diffraction (XRD). The XRD patterns of three DyF_3 nanosized samples are shown on Figure 2. They were recorded using a Bruker D8 Advance X-ray diffractometer with use of copper Kα

(α = 1.5418 A°) radiation at a scanning rate of $5^0 min^{-1}$ in 2θ range of 20–60^0 by step 0.015^0. Diffraction peaks could be indexed from the simulated pattern calculated by PowderCell [11] software (space group Pnma ((№62), lattice constants a = 0.6460 nm, b = 0.6906 nm, c = 0.4376 nm [12]). Obviously, that peaks on XRD pattern for samples #1-3 become narrower with increasing microwave-assisted hydrothermal treatment time (Figure 2a-2c).

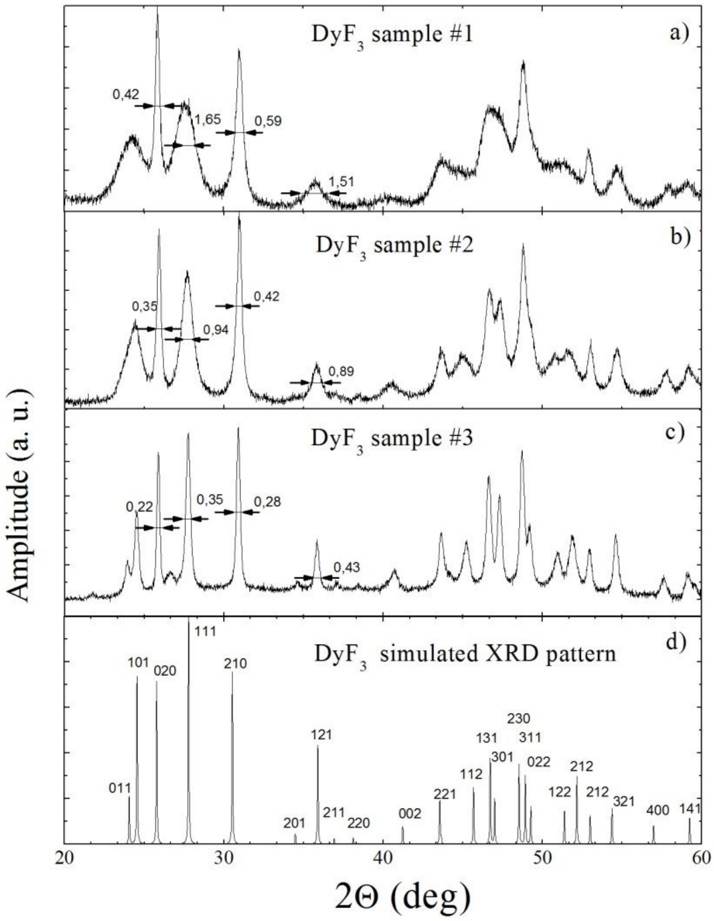

Figure 2. a) - c) experimental XRD patterns of synthesized DyF_3 nanosized samples #1-3. d) simulated XRD patterns in PowderCell software.

Thus, for the first time using a microwave hydrothermal method the series of nanoparticles DyF_3 were synthesized with average size about 16-18 nm. Influence of microwave radiation has been found on the particle size distribution and XRD peaks width.

Also the results of the synthesis nanoparticles DyF_3 and TbF_3 will be presented.

Acknowledgments

This work was partially supported by RNF grant №16-12-10359.

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