Data Article

Data supporting the results of the characterization of the phases and structures appearing during the synthesis process of \(\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Zn}_{2-x}\text{Ni}_x\text{Fe}_{12}\text{O}_{22}\) by auto-combustion

Tatyana Koutzarova\(^a\), Svetoslav Kolev\(^a\), Kiril Krezhov\(^a\), Borislava Georgieva\(^{a,*}\), Chavdar Ghelev\(^a\), Daniela Kovacheva\(^b\), Benedicte Vertruyen\(^c\), Raphael Closset\(^c\), Lan Maria Tran\(^d\), Michal Babij\(^d\), Andrzej J. Zaleski\(^d\)

\(^a\) Institute of Electronics, Bulgarian Academy of Sciences, 72 Tsarigradsko Chaussee, 1784 Sofia, Bulgaria
\(^b\) Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences, Acad. Georgi Bonchev Str., bld. 11, 1113 Sofia, Bulgaria
\(^c\) Greenmat, Chemistry Department, University of Liege, 11 allée du 6 août, 4000 Liège, Belgium
\(^d\) Institute of Low Temperature and Structure Research, Polish Academy of Sciences, Ul. Okólna 2, 50–422 Wroclaw, Poland

A R T I C L E   I N F O

Article history:
Received 5 May 2020
Revised 20 May 2020
Accepted 22 May 2020
Available online 02 June 2020

Keywords:
Y-type hexaferrite
Powder annealing
Crystalline size
Cation ratio
Auto-combustion technique

A B S T R A C T

The data presented has to do with identifying the various phases arising during the synthesis of the Y-type hexaferrite series \(\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Zn}_{2-x}\text{Ni}_x\text{Fe}_{12}\text{O}_{22}\) by auto-combustion that we deem important for their microstructural and magnetic properties. The data and the related analyses support the research paper “Ni-substitution effect on the properties of \(\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Zn}_{2-x}\text{Ni}_x\text{Fe}_{12}\text{O}_{22}\) powders” \([1]\). Thus, the parameters are presented of the phases appearing after auto-combustion and after the initial annealing at 800 °C, namely, crystal cell and crystallite size. Also, additional data are provided obtained by EDS concerning the \(\text{Ba}:\text{Sr}:\text{Zn}:\text{Ni}:\text{Fe}\) ratio in \(\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Zn}_{2.0-x}\text{Ni}_x\text{Fe}_{12}\text{O}_{22}\) \((x = 0.8, 1, 1.5)\) samples synthesized at 1170 °C for 10 h. The data can be used as a reference in establishing how the phases distinguished during the initial process of auto-combustion affect the \(\text{Ba}_{0.5}\text{Sr}_{1.5}\text{Zn}_{2-x}\text{Ni}_x\text{Fe}_{12}\text{O}_{22}\) powders, which are candidates for

\(*\) Corresponding author.
E-mail address: b.georgiewa@abv.bg (B. Georgieva).

https://doi.org/10.1016/j.dib.2020.105803
2352-3409/\© 2020 The Author(s). Published by Elsevier Inc. This is an open access article under the CC BY license.
(http://creativecommons.org/licenses/by/4.0/)
room-temperature multiferroic materials. The data have not been published previously and are made available to permit critical or further analyses.

© 2020 The Author(s). Published by Elsevier Inc. This is an open access article under the CC BY license. (http://creativecommons.org/licenses/by/4.0/)

Specifications Table

| Subject                  | Materials Science, Electronic, Optical and Magnetic Materials |
|--------------------------|---------------------------------------------------------------|
| Specific subject area    | Multiferroic Materials, Hexaferrites, Sol-Gel Auto-Combustion|
| Type of data             | Tables                                                         |
|                          | Figures                                                        |
|                          | Text file                                                      |
| How data were acquired   | X-ray diffraction (XRD) measurements performed using a Brucker D8 diffractometer. Scanning electron microscopy and energy dispersive X-ray spectroscopy (FEI XL30 FEG-ESEM, Bruker Quantax EDS coupled to ESEM). |
| Data format              | Raw                                                           |
|                          | Analyzed                                                      |
|                          | Filtered                                                      |
| Parameters for data collection | The characterization was implemented by X-ray diffraction (XRD) carried out by a Brucker D8 diffractometer (40 kV, 30 mA) controlled by DIFFRACTPLUS software in Bragg-Brentano reflection geometry with Cu-Kα radiation (λ = 1.5418 Å); and by scanning electron microscopy and energy dispersive X-ray spectroscopy (FEI XL30 FEG-ESEM, Bruker Quantax EDS coupled to ESEM). |
| Description of data collection | The XRD experiments were conducted on powder samples. The percentage, crystal cell parameters and crystallite size of the phases were determined from X-ray diffractograms. The EDS analyses were performed on four points on polished cross-sections of bulk samples (pellets) in view of finding the Ba: Sr: Zn: Ni: Fe ratio. |
| Data source location     | Institution: Institute of Electronics, Bulgarian Academy of Sciences |
|                          | City: Sofia                                                   |
|                          | Country: Bulgaria                                            |
|                          | Institution: Institute of General and Inorganic Chemistry     |
|                          | City: Sofia                                                   |
|                          | Country: Bulgaria                                            |
|                          | Institution: Greenmat, Chemistry Department, University of Liège, |
|                          | City: Liège                                                   |
|                          | Country: Belgium                                             |
| Data accessibility       | With the article                                             |
| Related research article | T. Koutzarova, S. Kolev, K. Krezhov, B. Georgieva, Ch. Ghelev, D. Kovacheva, B. Vertruyen, R. Closset, L. M. Tran, M. Babij, A. J. Zaleski, Ni-substitution effect on the properties of Ba_{0.5}Sr_{1.5}Zn_{2-x}Ni_{x}Fe_{12}O_{22} powders, J. Magn. Magn. Mater. doi: 10.1016/j.jmmm.2020.166725 |

Value of the Data

• The data distinguishes the intermediate phases appearing during the synthesis of a Y-type hexaferrite (Ba_{0.5}Sr_{1.5}Zn_{2-x}Ni_{x}Fe_{12}O_{22}) by auto-combustion;

• The EDS analysis yields reliable information on the chemical composition at different points on bulk Ba_{0.5}Sr_{1.5}Zn_{2-x}Ni_{x}Fe_{12}O_{22} samples;

• The data could be useful in identifying the phases and their effect during the synthesis of other hexaferrites;

• The data supplies important additional information to the related research article.
Table 1
Phase percentage, crystal cell parameters and average crystallite size obtained from the XRD patterns of auto-combusted powders and powder annealed at 800 °C for three hours.

| Sample                        | Spinel | (Ba,Sr)CO₃      | (Ba,Sr)FeO₃₋ₓ | BaSrFe₂O₈ |
|-------------------------------|--------|-----------------|----------------|------------|
| Ba₀.₅Sr₁.₅Zn₁₂Ni₀₈Fe₁₂O₂₂     |        |                 |                |            |
| auto-combusted powder         |        |                 |                |            |
| percentage                    | 87%    | 5.099(4) Å      | 5.664(1) Å     | 5.435(5) Å |
| a                             | 8.378(6) Å | 8.624(9) Å     | 21.681(4) Å   | 8.055(5) Å |
| b                             | 11 nm  | 6.098(4) Å      | 5 nm (fix)     | 79 nm      |
| c                             | 19 nm  |                 |                |            |
| crystallite size              |        |                 |                |            |
| Ba₀.₅Sr₁.₅Zn₁₂Ni₀₈Fe₁₂O₂₂     |        |                 |                |            |
| annealed at 800°C             |        |                 |                |            |
| percentage                    | 65%    | 5.685(4) Å      | 5.446(2) Å     |            |
| a                             | 8.395(5) Å | 6.105(5) Å     | 21.860(3) Å   |            |
| b                             | 11 nm  | 6.097(6) Å      | 5 nm (fix)     |            |
| c                             | 19 nm  |                 |                |            |
| crystallite size              |        |                 |                |            |
| Ba₀.₅Sr₁.₅Zn₁₂Ni₁₀₀Fe₁₂O₂₂    |        |                 |                |            |
| auto-combusted powder         |        |                 |                |            |
| percentage                    | 87%    | 5.101(0) Å      | 5.109(3) Å     | 5.434(5) Å |
| a                             | 8.371(1) Å | 8.621(8) Å     | 5.665(1) Å    | 8.055(5) Å |
| b                             | 11 nm  | 6.105(5) Å      | 21.860(3) Å   | 79 nm      |
| c                             | 27%    | 5.097(4) Å      | 19 nm (fix)    |            |
| crystallite size              |        |                 |                |            |
| Ba₀.₅Sr₁.₅Zn₀₉₀Ni₁₀₀Fe₁₂O₂₂   |        |                 |                |            |
| nitrate of spinel             |        |                 |                |            |
| percentage                    | 68%    | 5.097(4) Å      | 6.097(6) Å     | 8.601(6) Å |
| a                             | 8.385(5) Å | 6.097(6) Å     | 21.860(3) Å   | 79 nm      |
| b                             | 12 nm  | 6.097(6) Å      | 5 nm (fix)     |            |
| c                             | 19 nm  |                 |                |            |
| crystallite size              |        |                 |                |            |
| Ba₀.₅Sr₁.₅Zn₀₉₀Ni₁₀₀Fe₁₂O₂₂   |        |                 |                |            |
| nitrate of spinel             |        |                 |                |            |
| percentage                    | 27%    | 5.101(0) Å      | 5.109(3) Å     | 5.434(5) Å |
| a                             | 8.357(8) Å | 8.601(6) Å     | 21.860(3) Å   | 79 nm      |
| b                             | 9 nm   | 6.097(6) Å      | 5 nm (fix)     |            |
| c                             | 6%     | 5.097(4) Å      | 19 nm (fix)    |            |
| crystallite size              |        |                 |                |            |
| Ba₀.₅Sr₁.₅Zn₀₉₀Ni₁₀₀Fe₁₂O₂₂   |        |                 |                |            |
| nitrate of spinel             |        |                 |                |            |
| percentage                    | 67%    | 6.097(6) Å      | 79 nm          |            |
| a                             | 8.368(1) Å | 6.097(6) Å     | 21.740(4) Å   |            |
| b                             | 10 nm  | 6.097(6) Å      | 5 nm (fix)     |            |
| c                             | 19 nm  |                 |                |            |
| crystallite size              |        |                 |                |            |

1. Data description

The data and analyses included here corroborate the results of and the conclusions drawn from the study of Ba₀.₅Sr₁.₅Zn₂₋ₓNiₓFe₁₂O₂₂ (x = 0.8, 1, and 1.5) hexaferrites synthesized by sol-gel auto-combustion [1]. The XRD analysis was employed to distinguish between the phases formed during the consecutive steps of the synthesis of Ba₀.₅Sr₁.₅Zn₂₋ₓNiₓFe₁₂O₂₂ (x = 0.8, 1, and 1.5). Fig. 1 shows the XRD patterns of the auto-combusted powders and the powder treated at 800 °C for three hours for samples with x=1, and 1.5. The corresponding XRD patterns for sample Ba₀.₅Sr₁.₅Zn₁₂Ni₀₈Fe₁₂O₂₂ are presented in Koutzarova et al. [1]. A spinel-type phase (Ni-Zn mixed ferrite (Zn, Ni)Fe₂O₄) and (Ba, Sr)CO₃ were observed in the as-synthesized auto-combustion powders. The XRD-patterns of the powders heat-treated at 800 °C exhibit the peaks of spinel type phase BaFeO₃₋ₓ and BaSrFe₂O₈. Table 1 summarizes the data for the crystal cell.
Fig. 1. XRD-patterns of auto-combusted powders (a, c) and powder annealed at 800 °C for three hours (b, d) for samples $x = 1$ (a, b), and 1.5 (c, d).
Table 2
The Ba:Sr:Zn: Ni: Fe ratio estimated from the EDX analyses of Ba$_0.5$Sr$_{1.5}$Zn$_{1.2}$Ni$_{0.8}$Fe$_{12}$O$_{22}$ ($x = 0.8, 1, 1.5$) samples at the four points on polished cross-sections surface.

| Sample | Ba$_0.5$Sr$_{1.5}$Zn$_{1.2}$Ni$_{0.8}$Fe$_{12}$O$_{22}$ | Ba$_0.5$Sr$_{1.5}$ZnNiFe$_{12}$O$_{22}$ | Ba$_0.5$Sr$_{1.5}$Zn$_{0.5}$Ni$_{1.5}$Fe$_{12}$O$_{22}$ |
|--------|-------------------------------------------------|---------------------------------|---------------------------------|
| Point 1 | $0.5:1.5:11:0.8:12.3$ | $0.5:1.7:1.7:1.7:16.7$ | $0.5:1.5:0.5:14:12.9$ |
| Point 2 | $0.5:1.6:12:0.7:12.4$ | $0.5:1.5:1:8:1.5:16.6$ | $0.5:1.5:0.5:13:12.0$ |
| Point 3 | $0.5:1.5:11:0.8:12.4$ | $0.5:1.7:1.1:1.0:12.3$ | $0.5:1.7:0.5:13:12.6$ |
| Point 4 | $0.5:1.5:10:0.8:12.2$ | $0.5:1.6:2:2.2:15.1$ | $0.5:1.6:0.6:14:12.9$ |

parameters, the average crystallite size and the phases' percentage content derived from the XRD patterns. The original output files for all three samples ($x = 0.8, 1, 1.5$) obtained by Topas 4.2 are given in the supplementary file.

Fig. 2 displays XRD-patterns in the 2-theta range 37.3° – 45.3° of Ba$_0.5$Sr$_{1.5}$Zn$_{1.2}$Ni$_{0.8}$Fe$_{12}$O$_{22}$, Ba$_0.5$Sr$_{1.5}$ZnNiFe$_{12}$O$_{22}$ and Ba$_0.5$Sr$_{1.5}$Zn$_{0.5}$Ni$_{1.5}$Fe$_{12}$O$_{22}$ treated at 1170 °C. No signs were observed of nickel spinel ferrite decomposition to NiO during the Y-type hexaferrite formation process [2].

The statistical data yielded by the analysis of the four points of EDX spectrum of polished cross-sections of the bulk samples of Ba$_0.5$Sr$_{1.5}$Zn$_{1.2}$Ni$_{0.8}$Fe$_{12}$O$_{22}$, Ba$_0.5$Sr$_{1.5}$ZnNiFe$_{12}$O$_{22}$ and Ba$_0.5$Sr$_{1.5}$Zn$_{0.5}$Ni$_{1.5}$Fe$_{12}$O$_{22}$ are given in Table 2.

2. Experimental design, materials, and methods

Polycrystalline samples of Ba$_0.5$Sr$_{1.5}$Zn$_{2-x}$Ni$_{x}$Fe$_{12}$O$_{22}$ ($x = 0.8, 1, 1.5$) were fabricated by a modified citric acid sol-gel auto-combustion using stoichiometric amounts of the precursors; a detailed description of the sample preparation methodology is given in [1]. In brief, the powders produced after the auto-combustion process were annealed at 800 °C for three hours. All powders were subjected to homogenization by vibrating ball milling; then the resulting powders were pressed at 7 MPa to bulk pellets with a diameter of 16 mm. The pellets were heat-treated at 1170 °C in air for 10 h to obtain the Ba$_0.5$Sr$_{1.5}$Zn$_{2-x}$Ni$_{x}$Fe$_{12}$O$_{22}$ compositions with $x = 0.8, 1, 1.5$. Subsequently, the bulk pellets were cut and polished for microscopic studies.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships which have, or could be perceived to have, influenced the work reported in this article.

Acknowledgments

The work was supported partially by the Bulgarian National Science Fund under contract DN 08/4 “Novel functional ferrites-based magneto-electric structures”, by a joint research project between the Bulgarian Academy of Sciences and WBI, Belgium, and by a joint research project between the Bulgarian Academy of Sciences and the Institute of Low Temperature and Structure Research, Polish Academy of Sciences. B. Georgieva was supported for the synthesis and structural properties by the Bulgarian Ministry of Education and Science under the National Research Program “Young scientists and postdoctoral students” approved by DCM # 577/17.08.2018.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.dib.2020.105803.
Fig. 2. XRD-patterns of (a) Ba$_{0.5}$Sr$_{1.5}$Zn$_{1.2}$Ni$_{0.8}$Fe$_{12}$O$_{22}$, (b) Ba$_{0.5}$Sr$_{1.5}$ZnNi$_{1.2}$Fe$_{12}$O$_{22}$ and (c) Ba$_{0.5}$Sr$_{1.5}$Zn$_{0.5}$Ni$_{1.5}$Fe$_{12}$O$_{22}$ in the 2-theta range $37.3^\circ - 45.3^\circ$. The expected peak positions of NiO (PDF number 00–047–1049) are marked in red.
References

[1] T. Koutzarova, S. Kolev, K. Krezhov, B. Georgieva, Ch. Ghelev, D. Kovacheva, B. Vertruyen, R. Closset, L.M. Tran, M. Babij, A.J. Zaleski, Ni-substitution effect on the properties of Ba_{0.5}Sr_{1.5}Zn_{2-x}Ni_{x}Fe_{12}O_{22} powders, J. Magn. Magn. Mater. 505 (2020) 166725 https://doi.org/10.1016/j.jmmm.2020.166725.

[2] S. Corso, Ph. Tailhades, I. Pasquet, A. Rousset, V. Laurent, A. Gabriel, C. Condolf, Preparation conditions of pure and stoichiometric Ni_{x}Fe_{3-x}O_{4} bulk ceramics, Solid State Sci 6 (2004) 791–798 https://doi.org/10.1016/j.solidstatesciences.2004.03.037.