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X-RAY DIFFRACTION AND MÖSSBAUER SPECTROSCOPY INVESTIGATIONS OF THE (Al, Ni, Co)-DOPED AgFeO$_2$ SYNTHESIZED BY HYDROTHERMAL METHOD

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Abstract. *Delafossite AgFeO$_2$, AgFe$_{0.9}$Al$_{0.1}$O$_2$, AgFe$_{0.9}$Ni$_{0.1}$O$_2$, and AgFe$_{0.9}$Co$_{0.1}$O$_2$ powders were synthesized by hydrothermal method. The structural analysis and hyperfine interactions investigations were performed by X-ray diffraction and the Mössbauer spectroscopy. It was found that the (Al, Ni, Co)-doped delafossite phases with traces of metallic silver can be obtained during hydrothermal synthesis. Investigations revealed that the type of the incorporated element has an impact on the structural properties of the obtained delafossites. However, doping of cobalt, nickel, and alumina ions to the AgFeO$_2$ delafossite structure does not cause significant changes in the values of the hyperfine interactions parameters. The of the Mössbauer spectra confirm the paramagnetic character of the obtained compounds at room temperature.*

Keywords: delafossites, hydrothermal synthesis, X-ray diffraction, Mössbauer spectroscopy

BADANIA METODĄ DYFRAKCJI PROMIENIOWANIA X ORAZ SPEKTROSKOPII EFEKTU MÖSSBAUERA AgFeO$_2$ DOMIESZKOWANEGO JONAMI Co, Ni i Al WYTWARZANEGO METODĄ HYDROTERMALNĄ

Streszczenie. *Proszkowe próbki delafosytów AgFeO$_2$, AgFe$_{0.9}$Al$_{0.1}$O$_2$, AgFe$_{0.9}$Ni$_{0.1}$O$_2$, and AgFe$_{0.9}$Co$_{0.1}$O$_2$ zostały wytworzone metodą hydrotermalną. Badania pozwalające na analizę strukturalną oraz oszacowanie parametrów oddziaływań nadsubtelnych przeprowadzono z wykorzystaniem dyfrakcji rentgenowskiej oraz spektroskopii efektu Mössbauera. Udowodniono, że za pomocą metody hydrotermalnej istnieje możliwość wytworzenia delafosytu AgFeO$_2$ domieszkowanego jonami glinu, niklu i kobaltu z niewielką ilością zanieczyszczeń metalicznym srebrem. Badania wykazały, że rodzaj domieszki ma wpływ na właściwości strukturalne otrzymanych materiałów. Domieszkowanie jednak nie wpłynęło znacząco na zmiany wartości parametrów oddziaływań nadsubtelnych. Kształt zarejestrowanych widm mössbauerowskich potwierdza paramagnetyzm otrzymanych materiałów w temperaturze pokojowej.*

Słowa kluczowe: delafosyty, synteza hydrotermalna, dyfrakcja rentgenowska, spektroskopia mössabuerowska

Introduction

Delafossites are an interesting class of materials with a wide range of physical properties due to a variety of chemical compositions. They are described by the general formula of ABO$_2$, where A denotes monovalent cation of the *semi*-noble or the noble metal, i.e., Ag, Cu, Pd, or Pt. Site B may be occupied by trivalent cation of a. transition metal element (e.g., Cr, Fe, Mn, Ni, Co) or b. *p*-block element (e.g., Al, Ga, In, Tl) or c. rare earth element (e.g., Sc, La, Nd, Eu) [11]. Delafossite structure is depicted as stacking of two alternated layers. The first one consists of slightly distorted edge-shared B^{III}O$_6$ octahedra, the latter one is a monovalent layer of the closed-packed A^I ions. Linear coordination of A^I by two oxygen atoms along the *c*-axis leads to interlinkage of the adjacent BO$_2$ layers, where each oxygen is coordinated by four cations (one in the A-position and three in the B-site). Considering the stacking pattern of the alternated layers, the delafossite structure can form two polytypes, i.e., rhombohedral 3R type (space group *R-3m*) and hexagonal 2H polytype (space group I6$_3$/*mmc*) [15]. To date, numerous ABO$_2$ compounds have been reported with high *p*-type conductivity (10^{-2}–10^2 S·cm^{-1}) and high optical transparency (50%–85%), dependent on their chemical compositions and film depositions method [16]. These delafossite oxides could play important role in diverse photoelectronic and photoelectrochemical applications, such as field electron emitters, light-emitting diodes, solar cells, photocatalysts, functional windows, etc. [1].

Recently, much attention has been paid to developing new magnetic materials, e.g., multiferroics, diluted magnetic semiconductors, or magneto-optic materials [8, 13]. Delafossites also seem to be promising candidates for these purposes. Iron-based compounds, with the general formula AFeO$_2$ (where A denotes Ag or Cu elements), exhibit quite exotic magnetic properties. Below the Néel temperature, they have a complicated magnetic structure caused by frustrated exchange interactions induced by the triangular lattice system of Fe ions. This type of spins arrangement is responsible for inducing ferroelectric polarization, which can be described by the inverse Dzyaloshinskii-Moriya effect [9]. It is the reason, that both iron-based compounds, AgFeO$_2$, and CuFeO$_2$ are identified as multiferroics in the low temperature regime. Nevertheless, due to difficulties with the preparation of high-quality samples, the explaining of the iron-based delafossites properties is still the open question.

Modification of the delafossite properties can be realized by the ion substitution of trivalent B^{3+} cation [12, 19]. Recently, it was reported that the nickel doping of silver ferrite enhances significantly the AgFeO$_2$ catalytic activity [21]. Likewise, the cobalt doping in copper delafossites induces interesting phenomena. Dong et. al synthesized thin films of cobalt doped transparent CuAlO$_2$ semiconductors which exhibited weak ferromagnetism at room temperature [3]. Elkhouni et al. presented several papers devoted to the cobalt substitution of the CuCrO$_2$ and proved that the incorporation of the magnetic ions can induce new spin ordering and enhance magnetization [5, 6]. Wheatley et al. showed that the addition of the diluted amounts of Al to the CuFeO$_2$ host greatly improves samples conductivity [20].

To our knowledge, studies of AgFeO$_2$ delafossite doped with different ions at Fe position have been carried out only by several researchers (e.g., doping Cr to AgFeO$_2$ reported in [4]). Therefore, the lack of experimental data motivates further studies on the incorporation of the Al, Ni, and Co ions into the delafossite structure. Recognizing the structural properties of the doped AgFeO$_2$ will enrich knowledge about new delafossite family members and give information over possibilities of modifications of their physical properties in purpose to obtain advanced materials.

In this work, we synthesized AgFeO$_2$, AgFe$_{0.9}$Al$_{0.1}$O$_2$, AgFe$_{0.9}$Ni$_{0.1}$O$_2$, and AgFe$_{0.9}$Co$_{0.1}$O$_2$ samples by the hydrothermal method. The main goal of this study was to determine the impact of doping on the structural properties and hyperfine interactions parameters. The materials were characterized by X-ray diffraction and Mössbauer spectroscopy.

1. Experimental methods

The samples of AgFeO$_2$, AgFe$_{0.9}$Al$_{0.1}$O$_2$, AgFe$_{0.9}$Ni$_{0.1}$O$_2$, and AgFe$_{0.9}$Co$_{0.1}$O$_2$ were synthesized by the hydrothermal method. All of the chemicals were purchased with analytical grade

and used without further purification. As started reagents nitrates $Ag(NO_3)$, $Fe(NO_3)_3 \cdot 9H_2O$, $Al(NO_3)_3 \cdot 9H_2O$, $Ni(NO_3)_2 \cdot 6H_2O$, $Co(NO_3)_2 \cdot 6H_2O$, and NaOH were utilized. Firstly, the pure silver ferrite was synthesized. The iron and silver nitrates were dissolved in distilled water in the molar ratio of 1:1. Then, the NaOH solution was added slowly until the pH~12 was reached. Afterward, the obtained solution was stirred for 15 minutes to obtain a homologous precursor. Then the prepared precursor was transferred into a 100-mL Teflon-lined stainless-steel autoclave and placed into an oven at 180°C for 24 h. After cooling to room temperature, the brownish precipitate was collected and washed with deionized water and absolute ethanol several times. Finally, the product was dried in an oven at 180°C overnight to achieve $AgFeO_2$ powder. For the purpose to obtain $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ similar procedure was employed. The only difference was in the molar ratios of the starting reagents, which followed the formulas of the compounds listed above.

X-ray diffraction (XRD) studies were performed using a PanAlitical X'Pert Pro diffractometer working with a Cu lamp. For the phase and structural analysis, the X'Pert HighScore Plus software equipped with the ICDD PDF2 database was used. [57]Mössbauer spectra (MS) were registered at room temperature using a Polon spectrometer working in a transmission geometry and at constant acceleration mode. As a source of 14.4 keV gamma radiation of the [57]Co in a rhodium matrix was used. All values of the isomer shift within this paper are related to the α-Fe standard.

2. Results and discussion

Figure 1 shows the XRD patterns of $AgFeO_2$, $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ powders registered at room temperature. In the case of an un-doped sample of $AgFeO_2$, in the pattern only peaks characteristic for the silver ferrite phase are visible. It confirmed the purity of the delafossite phase. Comparison of the results with database standards corresponding to the hexagonal polytype $2H$-$AgFeO_2$ (PDF2 Card No. 01-075-2147) and rhombohedral $3R$-$AgFeO_2$ (PDF2 Card No. 00-029-1141) proved that the mixture of both phases was obtained. The lattice parameters for the un-doped sample were determined from the diffractograms, and they were as follows: $a = 3.039(1)$ Å and $c = 12.390(1)$ Å for $2H$ polytype, and $a = 3.039(1)$ Å and $c = 18.588(5)$ Å for $3R$ polytype. Parameters are comparable for all studied Al, Ni, and Co-doped silver ferrites. Their values are slightly higher in comparison to the results obtained in our work for $AgFeO_2$ synthesized by the co-precipitation method [17]. Moreover, the significant anisotropic broadening of diffraction lines observed in the case of co-precipitated materials is not seen now in XRD patterns registered for $AgFe_{0.9}Al_{0.1}O_2$ and $AgFe_{0.9}Co_{0.1}O_2$ (Fig. 1). It can mean a better level of crystallinity of the hydrothermally synthesized materials and a lower amount of defects in the crystalline structure in comparison to co-precipitated powder samples of $AgFeO_2$.

In the case of $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ samples additional peaks, marked as metallic silver, are visible in the diffractograms (Fig.1). Similarly, as it was reported in [17] for the $AgFe_{1-x}Co_xO_2$ (x = 0–0.2) series of samples synthesized by the co-precipitation method there were no peaks indicated another impurity phase. The absence of the CoO, NiO, and Al_2O_3 oxides can prove that our attempt the replacing the Fe ions with Al, Ni, and Co ions in the delafossite structure was realized successfully.

Bringing the assumption that B^{3+} should replace Fe^{3+} ions during substitution, the appearance of metallic silver is quite unexpected. Nevertheless, because cobalt(II) nitrate and nickel(II) nitrate were used as starting chemicals during synthesis, we postulate that in our case trivalent iron ions were substituted by divalent cobalt and nickel ions. This assumption may explain the appearance of metallic silver as a secondary phase in the case of the $AgFe_{0.9}Ni_{0.1}O_2$ and $AgFe_{0.9}Co_{0.1}O_2$ samples. The lower valence of cobalt and nickel ions than Fe^{3+} may be the reason

for the emergence of oxygen vacancies in the octahedral layer, which at the same time causes the lack of oxygen in some linear bonds O-Ag-O. Similar interpretations were reported in [2, 7] where the substitutions of B-site trivalent ions in $CuBO_2$ delafossites by cations like Co^{2+}, Sn^{4+} induced the appearance of the secondary phases like CuO oxide during the synthesis process. Thus, obtained results suggest that the substituting ions were rather Co^{2+} and Ni^{2+} than trivalent ions and obtained powders are non-stoichiometric compounds with oxygen deficiency described by formulas $AgFe_{0.9}Ni_{0.1}O_{2-\delta}$ and $AgFe_{0.9}Co_{0.1}O_{2-\delta}$.
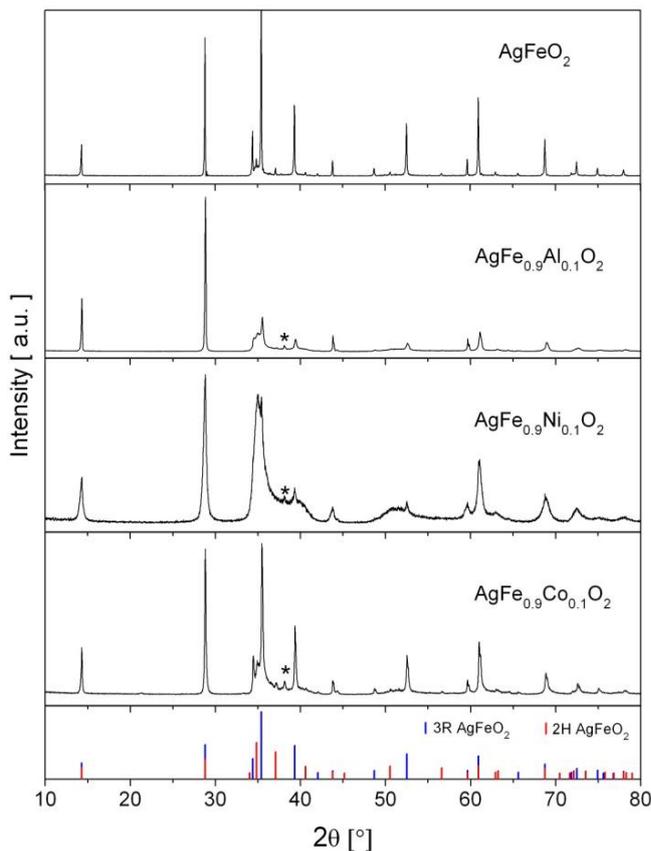
Fig. 1. XRD patterns registered for $AgFeO_2$, $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ samples; asterisks indicate metallic silver peak

Nevertheless, the above assumptions can not explain the appearance of the silver impurities in the case of $AgFe_{0.9}Al_{0.1}O_2$. The valence of both B-site ions is +3, so we can not assume the oxygen deficiencies. A possible explanation can be taken from Zwiener et. al [22]. They conducted comprehensive studies on the synthesis of $AgAlO_2$ under hydrothermal conditions and found that only optimized reaction conditions allow obtaining pure phase $AgAlO_2$ (temperature 210°C, reaction time 30 h). According to this work, the presence of the Ag by-product might be connected with the limited stability of the delafossite phase under the hydrothermal reaction conditions. Because of the lack of literature data confirmed these findings, we conclude that the appearance of the small amount of metallic silver in the $AgFe_{0.9}Al_{0.1}O_2$ sample might be connected with the complexity of the hydrothermal reaction in general and this problem needs further studies.

Moreover, the shapes of the diffractograms for particular samples are quite different. For $AgFeO_2$, diffraction lines are sharp and well separated. The original shape is more or less maintained for samples doped with nickel and cobalt ions, but the intensities of particular peaks are quite different. It may be connected with the appearance of some strains of the crystalline structure induced by the introduction to the structure ions with different values of radii at B position (Fe^{3+} – 0.645Å, Ni^{2+} – 0.69Å, Co^{2+} – 0.745Å, Al – 0.535Å) [14].

For the sample $AgFe_{0.9}Ni_{0.1}O_2$, significant anisotropic broadening of diffraction lines is observed. It may be seen that only peaks belonging to the (00*l*) family are sharp and well separated, but the peaks connected with the (10*l*) are much more broadened. It suggests that crystallite sizes ranged from a few to several dozen nm. Similar results were observed for co-precipitated $AgFeO_2$ in [18], where the shape of the spectra suggested that the powder particles have an elongated shape in the one crystallographic direction.

It is also worth mentioning that all the samples are a mixture of two polytypes, 2*H* and 3*R*. In the case of the un-doped sample, the polytype content was estimated to be as follows: 2*H* – 5%, and for 3*R* – 95%. Likewise, for the $AgFe_{0.9}Al_{0.1}O_2$ and $AgFe_{0.9}Co_{0.1}O_2$, the rhombohedral polytype is the dominating phase. However, for $AgFe_{0.9}Ni_{0.1}O_2$ it has been estimated that the ratio of the two polytypes is approximately 1:1. Hence, one can conclude, that the type of doped ions also has an impact on the phase composition of the obtained sample.
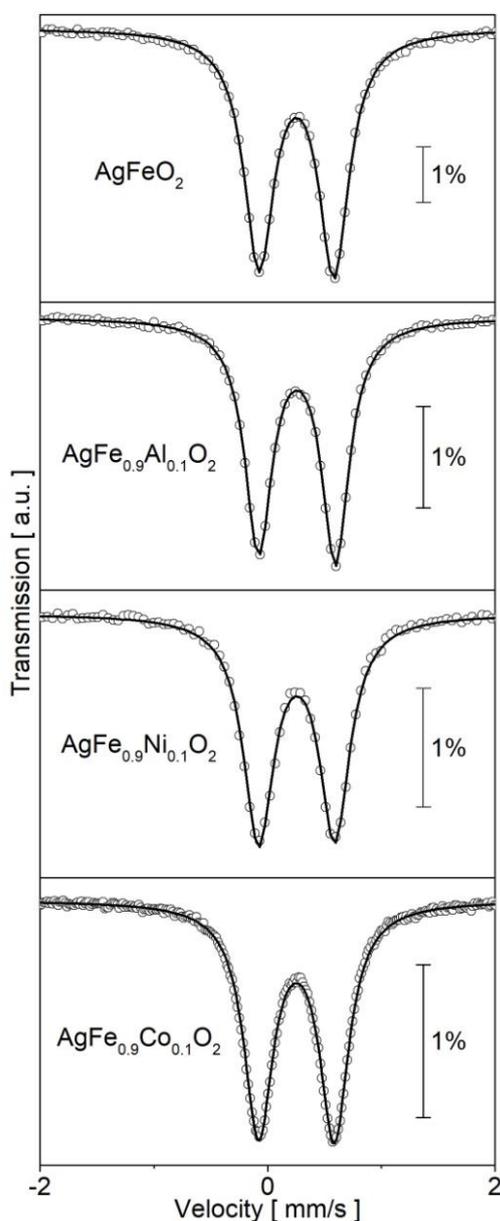
Fig. 2. Room-temperature Mössbauer spectra of the $AgFeO_2$, $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ samples

Results of the Mössbauer spectroscopy studies are presented in Fig. 2 and Table I. All the Mössbauer spectra registered at room temperature for the investigated samples consist of one quadrupole doublet corresponding to the delafossite phase. It confirms their paramagnetic character. The obtained hyperfine interactions parameters are summarized in Table 1. Values of isomer shifts (δ) and quadrupole splitting (Δ) are similar for all investigated samples and are in good agreement with literature data reported for the $AgFeO_2$ compound [10]. It is worth to mention that the numerical fitting of Mössbauer spectra was made by one component despite 3*R* and 2*H* polytypes coexisting in the material. This is because ^{57}Fe ions have the same nearest neighborhood in both structures, namely iron ions have 6 oxygen ions in the first, 6 iron ions in the second, and 6 silver ions in the third coordination sphere. Moreover, the interatomic distances between iron and the nearest neighbors are practically the same for both polytypes [18]. Thus, the well-resolved doublet with the value of the isomer shift 0.362(1) mm/s seen in MS spectra registered for un-doped sample confirms the existence of high-spin Fe^{3+} ions in an octahedral oxygen environment. The values of isomer shift determined for the $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ samples confirm that only high-spin Fe^{3+} ions in the octahedral coordination are present in the studied materials. Rather high values of the quadrupole splitting mean that a strong electric field gradient (EFG) occurs in the Fe^{3+} position in the crystalline lattice.

The width of the spectral lines Γ (half-width at half maximum) for the un-doped sample and doped samples are slightly higher from the natural width ($\Gamma = 0.12$ mm/s according to the certificate of the Mössbauer source manufactured by RITVERC GmbH). No distinct differences in the width lines values are seen. Hence, it may be surmised that the incorporation of the Al, Ni, and Co ions in the delafossite structure has random distribution.

Table 1. The results from the fitting of the Mössbauer spectra registered at room temperature: δ – isomer shift, Δ – quadrupole splitting, Γ – the width of spectral lines. The uncertainty of all values is 0.001 mm/s

Sample	δ [mm/s]	Δ [mm/s]	Γ [mm/s]	χ
$AgFeO_2$	0.362	0.658	0.159	0.89
$AgFe_{0.9}Al_{0.1}O_2$	0.366	0.676	0.145	0.95
$AgFe_{0.9}Ni_{0.1}O_2$	0.360	0.666	0.156	1.26
$AgFe_{0.9}Co_{0.1}O_2$	0.363	0.668	0.160	1.12

3. Conclusions

Delafossite $AgFeO_2$, $AgFe_{0.9}Al_{0.1}O_2$, $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$ fine powders were successfully synthesized by hydrothermal method. The XRD study proved the obtaining of (Al, Ni, Co)-doped delafossite phases with traces of metallic silver as the secondary phase. In the case of $AgFe_{0.9}Ni_{0.1}O_2$, and $AgFe_{0.9}Co_{0.1}O_2$, the lack of cobalt and nickel-containing secondary products suggests that Co^{2+} and Ni^{2+} substitute Fe^{3+} ions in delafossite lattice. The presence of metallic silver in the $AgFe_{0.9}Al_{0.1}O_2$ sample can be explained under the assumption of the complexity of the hydrothermal process. Moreover, some changes in the $AgFeO_2$ structure depending on the type of the incorporated ion were seen.

The Mössbauer spectroscopy studies revealed no distinct changes in the values of hyperfine interactions parameters. They confirm the paramagnetic character of the obtained samples at room temperature. Obtained results suggest that the hydrothermal synthesis causes the random distribution of the incorporated ions in the silver ferrite structure.

Acknowledgments

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