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MAGNETIC PROPERTIES OF HIGH-PRESSURE SYNTHESIZED

 $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) COBALTITES

(Communicated by Corresponding Member Valery M. Fedosyuk)

Abstract. The present study investigates the impact of high-pressure synthesis on the magnetic properties of 314-related $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) cobaltites. The observed magnetic behavior is driven by an increase in oxygen content, leading to the formation of a significant number of Co^{4+} ions. This results in enhanced ferromagnetic interactions through $\text{Co}^{4+}-\text{O}-\text{Co}^{3+}$ exchange and/or the emergence of superparamagnetic clusters below T_C , attributed to electron transfer between IS Co^{3+} and LS Co^{4+} . These findings highlight the role of oxygen stoichiometry in tuning the magnetic properties of perovskite-related cobaltites.

Keywords: cobaltites, high-pressure synthesis, crystal structure, magnetic properties

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МАГНИТНЫЕ СВОЙСТВА СИНТЕЗИРОВАННЫХ
ПОД ВЫСОКИМ ДАВЛЕНИЕМ КОБАЛЬТИТОВ $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$)

(Представлено членом-корреспондентом В. М. Федосюком)

Аннотация. Исследовано влияние синтеза под высоким давлением на магнитные свойства семейства 314 кобальтитов $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$). Наблюдаемое магнитное поведение обусловлено увеличением содержания кислорода, что приводит к образованию значительного количества ионов Co^{4+} . Как следствие, происходит усиление ферромагнитного состояния из-за возникновения сверхобменных взаимодействий $\text{Co}^{4+}-\text{O}-\text{Co}^{3+}$ и/или появления суперпарамагнитных кластеров ниже T_C , связанных с переносом электронов между IS Co^{3+} и LS Co^{4+} . Полученные результаты подчеркивают роль стехиометрии кислорода в формировании магнитных свойств перовскитоподобных кобальтитов.

Ключевые слова: кобальтиты, синтез под высоким давлением, кристаллическая структура, магнитные свойства

Для цитирования. Магнитные свойства синтезированных под высоким давлением кобальтитов $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) / Р. А. Лановский, О. С. Мантыцкая, Н. В. Терешко [и др.] // Доклады Национальной академии наук Беларуси. – 2025. – Т. 69, № 3. – С. 192–197. <https://doi.org/10.29235/1561-8323-2025-69-3-192-197>

Introduction. Layered cobaltites of the 314-cobaltite family (314 refers to the composition $\text{Sr}_3\text{LnCo}_4\text{O}_{10.5-d}$, where Ln represents a lanthanide) are an interesting class of materials that have been actively studied in recent years [1–5]. These materials exhibit G-type antiferromagnetic ordering with a small ferromagnetic component and a magnetic ordering temperature above room temperature. The ideal structure of layered 314 cobaltites contains a significant number of oxygen vacancies. It is well known that variations in oxygen content can dramatically affect physical properties of cobaltites.

It was shown that $\text{SrCoO}_{3-\gamma}$ can exhibit various structural distortions depending on the synthesis conditions and, consequently, on oxygen content: at $\gamma = 0.5$ it adopts an orthorhombic structure; at $\gamma = 0.15$ a tetragonal structure; and in the nearly stoichiometric case, a cubic structure with space group $\text{Pm}\bar{3}\text{m}$ [6].

In this paper, we report a study of the magnetic properties of a $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) cobaltites, which are related to the 314-cobaltite family. The samples were obtained using different synthesis methods, resulting in varying oxygen contents.

Materials and methods of research. Samples preparation. Polycrystalline samples of solid solutions $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) were synthesized using two different methods: conventional solid-state reaction in air conditions and high-pressure synthesis. The initial reagents of high purity Eu_2O_3 , SrCO_3 , Co_3O_4 and ZnO were taken in stoichiometric proportions and thoroughly mixed in a planetary ball mill RETSCH for 30 minutes at a speed of 250 rpm. To remove moisture, Eu_2O_3 was pre-annealed at 1000 °C for 2 hours before weighing.

A preliminary synthesis was carried out for 21–22 hours at a temperature of 1000 °C. The resulting material was then ground into a fine powder, which served as the precursor for the final synthesis.

For the conventional solid-state reaction method, the final synthesis was carried out at a temperature of 1150 °C for 8 hours with cooling for at least 10 hours down to a room temperature.

The second batch of the samples was synthesized under high-pressure conditions. Grounded powder of pre-synthesized sample was packed into catlinite form with a graphite heater. A pre-sintering step allows to avoid the decomposition of strontium carbonate, which would otherwise release gas in a closed volume during high-pressure synthesis. The sample was shielded from the graphite heated by molybdenum screen to prevent unwanted reaction of the oxygen-containing sample with the graphite heater. The synthesis was carried out at 1150–1250 °C under a pressure of 4.5 GPa for 3 minutes.

Instrumental details. Room temperature phase analysis of the samples was performed by X-ray powder diffraction (XRD) using an X-ray diffractometer DRON-3M (CuK-radiation) with a scanning step of 0.03° and an exposure time of 5 seconds. The data were refined using the Rietveld method [7] and the FullProf program [8]. As a basic function to describe the shape of the peaks, the Thompson–Cox–Hastings pseudo-Voigt function was chosen [9]. Due to the difficulty of detecting parameters associated with light elements such as oxygen using a conventional X-ray diffractometer, the oxygen Debye–Waller factor values were fixed during the refinement. The oxygen content was taken in accordance with the iodometry data, and the priority of filling certain oxygen positions was brought into line with the neutron diffraction data of the previously investigated Y-314 cobaltites [4].

Magnetic measurements were performed on a universal measuring device for physical properties (Cryogenic Ltd.) in magnetic fields up to 14 T and in the temperature range of 5 to 315 K.

Iodometry. Oxygen content was determined via iodometric titration. For each sample, 20–60 mg of powder was dissolved in dilute hydrochloric acid containing KI (always kept above metallic Zn and never deposit into air). Then the released iodine was titrated with $\text{Na}_2\text{S}_2\text{O}_3$. The titration end-point was detected using starch-indicator. Each sample of solid solutions was titrated up to 5 times to achieve high accuracy of oxygen index determination and to eliminate possible errors at one of the titration stages. In addition, a blank titration (without a sample) was carried out to ensure that atmospheric oxygen does not affect the results.

Results and discussion. Samples characterization. Based on the XRD data, it was found that all samples, whether synthesized conventionally or under high pressure, can be described using the tetragonal I4/mmm space group with $2a_p \times 2a_p \times 4a_p$ super cell with alternating layers of CoO_6 and $\text{CoO}_{4+\delta}$. The refined crystal structure includes three types of Sr/Eu atoms at Wyckoff sites 4e (0, 0, z) and 8g (0, 0.5, z), two types of Co atoms placed at 8h (x , x , 0) and 8f (0.25, 0.75, 0.25), and four different oxygen

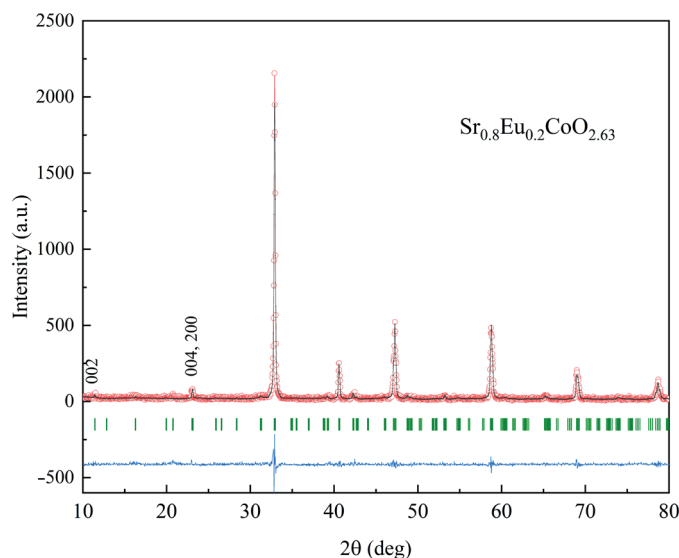


Fig. 1. Observed (circles), calculated (line) and difference patterns of XRD data of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$ at room temperature in model with space group $I4/mmm\ 2a_p \times 2a_p \times 4a_p$ super cell, conventional synthesis. The vertical bars indicate the calculated position of the nuclear Bragg peaks

Crystal structural parameters of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) solid solutions in model with space group $I4/mmm\ (2a_p \times 2a_p \times 4a_p)$ determined from the XRD data refinement, $T = 300$ K. High-pressure synthesized samples are marked with ‘HP’

Sample	$\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$	HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.83}$	$\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.62}$	HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.91}$
a, b (Å)	7.680 (27)	7.677 (49)	7.679 (45)	7.657 (34)
c (Å)	15.410 (20)	15.443 (19)	15.433 (88)	15.417 (98)
V (Å ³)	908.9 (94)	910.2 (82)	910.1 (96)	904.0 (32)
χ^2	1.56	1.73	1.71	1.68

positions, O1 16n (0, y, z), O2 16m (x, x, z), O3 8i (0, y, 0), and O4 8j (x, 0.5, 0). Figure 1 shows a result of processing the X-ray diffraction spectra of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$ synthesized by conventional solid state technique. The main crystal structural parameters of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) are summarized in Table.

It should be noted that for other 314-family cobaltites, the true symmetry has been identified as monoclinic $A2/m$ [2; 4]. However, the resolving power of a conventional laboratory diffractometer is insufficient to resolve these subtle differences. Nevertheless, a number of authors have successfully described similar 314 cobaltites using the tetragonal $I4/mmm$ space group with sufficient accuracy [1; 3–5; 10].

According to iodometry, the oxygen content significantly increased in samples synthesized under high pressure compared to those synthesized conventionally. This can be attributed to the higher oxygen content in the resulting precursor after the pre-synthesis stage. During subsequent synthesis under high pressure, the excess oxygen remains trapped in the closed high-pressure environment, leading to overoxidized samples.

Magnetometry. Figure 2 shows temperature dependences of magnetization obtained on heating after cooling without a field (ZFC) and in a field of 0.02 T (FC). Panels (a) and (b) correspond to conventionally synthesized samples, while (c) and (d) represent high-pressure synthesized samples. The isothermal magnetization curves at $T = 5$ K in a field up to 14 T are shown in Figure 3.

$\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$ and $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.62}$ exhibit magnetic ordering temperature higher than room temperature, typical for 314-cobaltites. A second inflection point is observed in the FC and ZFC curves at approximately 150 and 120 K, respectively (see figure 2, a, b). As clearly can be seen, high-pressure synthesized samples show significantly larger magnetization values (see figure 2, c, d).

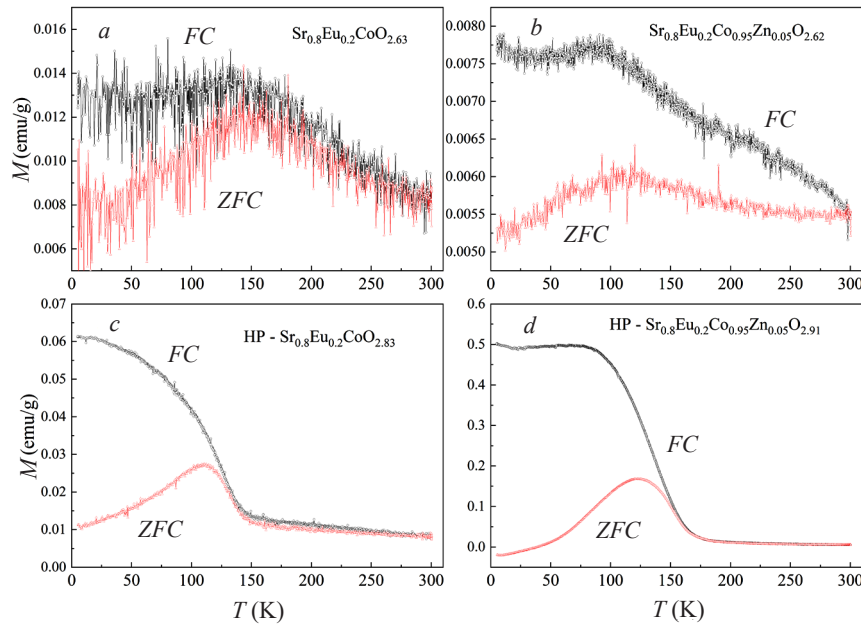


Fig. 2. Temperature dependences of the magnetization of the $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0, 0.05$, field cooling (FC) and zero field cooling (ZFC), measured at 0.02 T field). High-pressure synthesized samples are marked with ‘HP’

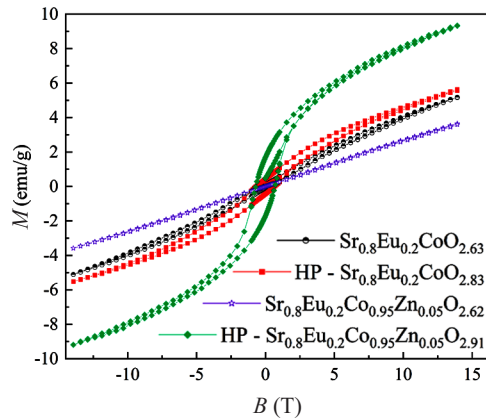


Fig. 3. Isothermal dependences of the magnetization of the $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0, 0.05$), $T = 5$ K. High-pressure synthesized samples are marked with ‘HP’

The coercive force of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$ is 0.21 T with a remanent magnetization of 0.12 emu/g at 5 K (see figure 3). With substitution of Co ions with Zn ions both coercive force and remanent magnetization drops, resulting in an almost linear field dependence for $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.62}$ and almost order of magnitude smaller magnetization values at temperature dependences of magnetization.

For high-pressure synthesized (are marked with HP) samples, HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.83}$ exhibits a coercive force of 0.69 T and a remanent magnetization of 0.44 emu/g at 5 K. Zn substitution in HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.91}$ results in a similar coercive force of 0.70 T but much larger remanent magnetization of 1.71 emu/g.

These drastic changes of magnetic properties can be attributed to an increase in oxygen content with switching from conventional synthesis to high-pressure synthesis. Formal oxidation state of Co ions of $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.63}$ is 1.06, corresponding to nearly exclusively Co^{3+} . For HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{CoO}_{2.83}$ the formal oxidation state is 1.46, denoting nearly 50/50 Co^{3+} to Co^{4+} ratio. For Zn-substituted $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.62}$ and HP- $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{0.95}\text{Zn}_{0.05}\text{O}_{2.91}$ the oxidation state is 1.116 and 1.705, respectively.

The 314-like cobaltites are G-type antiferromagnets with a small high-temperature ferromagnetic component, originating from the competition between positive and negative exchange interactions in anion-deficient layers, leading to the stabilization of canted magnetic structure [4].

The appearance of Co^{4+} ions, along with a decrease in oxygen vacancy concentration, favors positive $\text{Co}^{4+}\text{--O--Co}^{3+}$ exchange interactions, resulting in the observed changes in magnetic properties. Another possible explanation is based on the theory proposed by Goodenough, Zhou, and Yamaguchi et al. [11; 12] for the $\text{La}_{1-x}\text{Sr}_x\text{CoO}_3$ system. They suggested that the strong covalent bonding of the Co^{4+}O_6 LS complex stabilizes IS Co^{3+} at the nearest-neighbor cobalt, and ferromagnetic electron transfer from IS Co^{3+} to LS Co^{4+} gives rise to superparamagnetic clusters. The superparamagnetic clusters interact throw the Co^{3+} matrix to form a spin glass and with long-range ferromagnetic ordering below T_C .

Both of these scenarios are possible due to the formation of a large number of tetravalent cobalt ions as a result of sample overoxidation. This overoxidation leads to the dominance of above discussed mechanisms rather than the small ferromagnetic component associated with the canted non-collinear structure.

Conclusions. Our study shows that high-pressure synthesis results in pronounced changes in the magnetic properties of the 314-related $\text{Sr}_{0.8}\text{Eu}_{0.2}\text{Co}_{1-x}\text{Zn}_x\text{O}_{3-d}$ ($x = 0; 0.05$) cobaltites. These magnetic property changes can be attributed to an increase in oxygen content, which promotes the formation of a large number of Co^{4+} ions. The mechanism underlying the occurrence of a “more ferromagnetic” state may involve both the straightforward formation of positive $\text{Co}^{4+}\text{--O--Co}^{3+}$ exchange interactions and/or the formation of superparamagnetic clusters below T_C , due to the ferromagnetic electron transfer from IS Co^{3+} to LS Co^{4+} .

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