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Study and optimization of the synthesis routine of the single phase $\text{YBaCo}_2\text{O}_{6-\delta}$ double perovskite

The chemical interaction of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ with formation of double perovskite was studied depending on temperature and oxygen partial pressure. The stability of YCoO_3 was shown to have a crucial influence on the kinetics and mechanism of $\text{YBaCo}_2\text{O}_{6-\delta}$ formation. It was found that at 1000 °C in air, i.e. under conditions when YCoO_3 is unstable, the double perovskite $\text{YBaCo}_2\text{O}_{6-\delta}$ is formed much slower compared to the pure oxygen atmosphere where YCoO_3 is stable at the same temperature. Thus controlling YCoO_3 stability was shown to be the factor of key importance for optimal preparation of the $\text{YBaCo}_2\text{O}_{6-\delta}$ single phase.

Keywords: YBaCo_2O_6 synthesis, YCoO_3 instability, $p\text{O}_2$ acceleration, double perovskite synthesis, YBaCo_2O_5 , YCoO_3 , BaCoO_3 .

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Introduction

Complex oxide $\text{YBaCo}_2\text{O}_{6-\delta}$ with double perovskite structure has been extensively investigated in recent years as a promising material for oxygen membranes [1, 2] and solid oxide fuel cells (SOFCs) [3, 6, 7, 9, 12] due to high mixed ionic-electronic conductivity [12] and moderate thermal expansion comparable to that of the state-of-the art SOFC electrolytes [3]. However, $\text{YBaCo}_2\text{O}_{6-\delta}$ is unstable in air at temperatures between 800 and 850 °C [13] and decomposes to mixture of the simple perovskites $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$, which are more thermody-

namically stable under these conditions. This significantly impedes obtaining a single phase material. Moreover, a synthesis routine, which could be provided the single phase $\text{YBaCo}_2\text{O}_{6-\delta}$ obtaining, has not been discussed in literature so far. The lack of the appropriate data also inhibits a commercial application of the $\text{YBaCo}_2\text{O}_{6-\delta}$ based materials.

Therefore the main aim of the current work was to study a formation of the $\text{YBaCo}_2\text{O}_{6-\delta}$ double perovskite at 900 and 1000 °C in different gas atmospheres in order to optimize its synthesis routine.

Experimental

Taking into account that synthesis of $\text{YBaCo}_2\text{O}_{6-\delta}$ proceeds through the forma-

tion of intermediate phases of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ like other double perovs-

kites $\text{LnBaCo}_2\text{O}_{6-\delta}$ [14] as well as that a synthesis routine for these intermediate phases has been already described in literature [15, 16] we selected $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ as starting reagents for preparation of the $\text{YBaCo}_2\text{O}_{6-\delta}$ double perovskite.

Powder samples of $\text{YCoO}_{3-\delta}$, $\text{BaCoO}_{3-\delta}$ were synthesized by means of glycerol-nitrate technique, using Co, Y_2O_3 and BaCO_3 as starting materials. All the materials used had a purity of 99.99%. Metallic Co was obtained by reduction of Co_3O_4 (purity 99.99%) in H_2 atmosphere at 600 °C. Y_2O_3 and BaCO_3 were preliminarily calcined at 1100 °C and 600 °C, respectively, in air for two hours in order to remove adsorbed H_2O and CO_2 . Stoichiometric mixture of starting materials was dissolved in concentrated nitric acid. Then the required quantity of glycerol as a complexing and reducing agent was added to the obtained solution. Afterwards the solution was evaporated to dryness, and resulted dry powder was pyrolyzed. The product of pyrolysis was put in a cru-

cible and calcined in a furnace. The final calcination was carried out at 1100 °C in air for two hours C for $\text{BaCoO}_{3-\delta}$ and at 900 °C for $\text{YCoO}_{3-\delta}$. Phase composition of the as-prepared powder samples was confirmed by X-ray diffraction using Shimadzu XRD-7000 diffractometer (CuK α radiation, $20 \leq 2\theta, \leq 90$). X-ray diffraction patterns of the as-synthesized $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ are shown in Figs. 1 and 2.

The results of the structureless Le Bail fitting are also shown in Fig. 1 and 2. It should be noted that the X-ray diffraction pattern of BaCoO_3 was interpreted as a mixture of two compounds: BaCoO_3 and $\text{BaCoO}_{2.61}$ (see Fig. 2). The refined cell parameters of the prepared compounds given in Table 1 are in a good agreement with those reported in literature.

Synthesis of $\text{YBaCo}_2\text{O}_{6-\delta}$ was studied by annealing equimolar mixture of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ for 72h (6 steps with duration of 12 h at each step) at temperatures 900 and 1000 °C in atmospheres with oxygen partial pressure ($p\text{O}_2$)

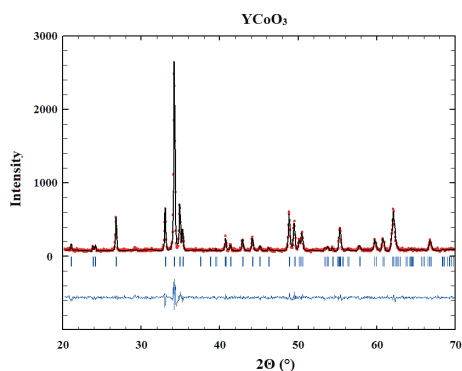


Fig. 1. X-ray diffraction pattern and its matching refinement plot of $\text{YCoO}_{3-\delta}$: observed X-ray diffraction intensity - points and calculated curve ($\chi^2 = 1.62$) - line. The bottom curve is the difference of patterns, $y_{\text{obs}} - y_{\text{cal}}$, and the small bars indicate the angular positions of the allowed Bragg reflections

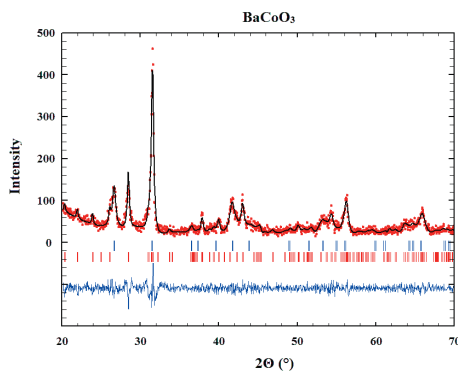


Fig. 2. X-ray diffraction pattern and its matching refinement plot of $\text{BaCoO}_{3-\delta}$: observed X-ray diffraction intensity - points and calculated curve ($\chi^2 = 1.87$) - line. The bottom curve is the difference of patterns, $y_{\text{obs}} - y_{\text{cal}}$, and the small bars indicate the angular positions of the allowed Bragg reflections for BaCoO_3 (blue lines) and $\text{BaCoO}_{2.61}$ (red lines)

0.21 and 1 atm with intermediate mixture regrinding in agate mortar. Phase compo-

sition of the samples after each step of annealing was controlled by XRD.

Table 1

Crystallographic parameters of synthesized cobaltites in comparison with literature data					
Compound	Space group	a^* , Å	b^* , Å	c^* , Å	Reference
YCoO _{3-δ}	Pbnm	5.139	5.419	7.365	this work
		5.137	5.420	7.364	[17]
		5.132	5.411	7.360	[18]
BaCoO ₃	P-6m2	5.683	5.683	4.552	this work
		5.645	5.645	4.752	[19]
		5.652	5.6525	4.763	[20]
BaCoO _{2.63}	P63/mmc	5.666	5.666	28.494	this work
		5.665	5.665	28.493	[16]
		5.671	5.671	28.545	[21]

* uncertainty ± 0.001 Å.

Results and discussion

Fig. 3 shows XRD patterns of the YCoO_{3-δ} + BaCoO_{3-δ} equimolar mixtures annealed at 900 °C in air ($p_{O_2} = 0.21$ atm) and pure oxygen ($p_{O_2} = 1$ atm) for 72 h. As seen annealing neither in air nor in oxygen atmosphere leads to formation of the single phase YBaCo₂O_{6-δ} at least for this time of annealing.

Moreover XRD pattern of the mixture annealed at 900 °C in pure oxygen atmosphere does not show any indication of the chemical interaction between the reagents

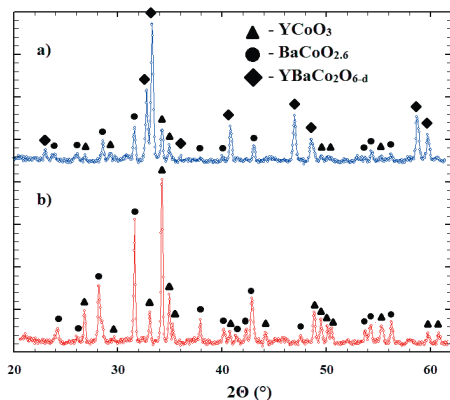


Fig. 3. XRD patterns of YCoO_{3-δ} and BaCoO_{3-δ} equimolar mixtures after annealing in air (a) and pure oxygen (b) at 900 °C for 72 h

and formation of YBaCo₂O_{6-δ} double perovskite whereas annealing in air leads to formation of significant amount of this double perovskite (see Fig. 3). Possible reason of this difference seems to be related to the instability of YBaCo₂O_{6-δ} oxide under oxidizing conditions at temperatures lower than some threshold value [11–13].

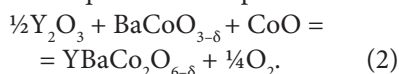
Figs. 4 and 5 show XRD patterns of the YCoO_{3-δ} and BaCoO_{3-δ} equimolar mixtures annealed at 1000 °C in air ($p_{O_2} = 0.21$ atm) and pure oxygen ($p_{O_2} = 1$ atm) for 72 h. As seen annealing in air also did not lead to the formation of the single phase double perovskite. Y₂O₃, BaCoO₃ and CoO can be identified as impurities in the X-ray diffraction pattern shown in Fig. 4. The presence of these impurities is a consequence of instability of the YCoO₃, which decomposes in air at $T \geq 900$ °C with formation of Y₂O₃ and CoO [11–13, 22, 23]. Similar behavior is well-known for the perovskite-type cobaltites with small rare-earth elements [24, 25].

Therefore formation of YBaCo₂O_{6-δ} at 1000 °C in air seems to proceed according

to the two-stage process. First YCoO_3 decomposes into Y_2O_3 and CoO upon heating of the equimolar mixture of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ up to 1000°C in air



Then a mixture of Y_2O_3 , BaCoO_3 and CoO slowly reacts at 1000°C with formation of the required double perovskite



At the same time annealing the $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ equimolar mixture at 1000°C in oxygen for 72 h leads to formation of the single phase $\text{YBaCo}_2\text{O}_{6-\delta}$ as seen in Fig. 5 where appropriate XRD pattern is shown. This pattern was refined as a mixture of two phases having $3\times 2\times 2$ and $1\times 2\times 2$ superstructures. The former has tetragonal structure (s.g. $P4/mmm$) with cell parameters $a = b = 11.596(4)$ Å and $c = 7.509(7)$ Å whereas the latter has orthorhombic structure (s.g. $Pmma$) with cell parameters $a = 3.821(4)$ Å, $b = 7.846(2)$ Å, $c = 7.515(8)$ Å in full agreement with available structural data [5, 7, 12, 26, 27].

Detailed step-by-step investigation of the $\text{YBaCo}_2\text{O}_{6-\delta}$ synthesis in oxygen at this temperature revealed that the result-

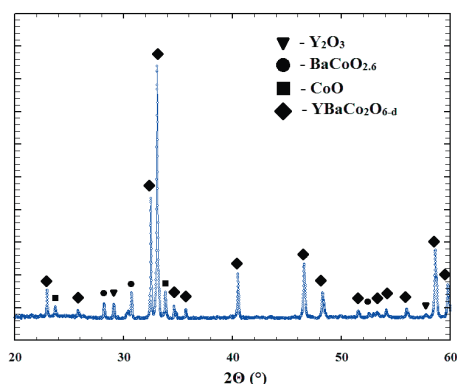


Fig. 4. XRD pattern of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ equimolar mixtures annealed in air at 1000°C for 72 h

ant mixture at each step except last one contained $\text{BaCoO}_{3-\delta}$, Y_2O_3 , CoO , $\text{YCoO}_{3-\delta}$ and the product $\text{YBaCo}_2\text{O}_{6-\delta}$. This result can be understood, first of all, based on the analysis of the thermodynamics of reaction Eq. (1). Although for this particular reaction thermodynamic functions are unknown similar reactions for Ho- and Er-contained cobaltites have already been studied in this respect [24, 25]. Required thermodynamic data for them are given in Table 2. As seen HoCoO_3 decomposition starts at 1051°C in air whereas ErCoO_3 decomposes already at 866°C in the same atmosphere. YCoO_3 as mentioned above is somewhere between these two compounds since its decomposition in air starts at $900\text{--}950^\circ\text{C}$ [11–13, 22, 23]. Therefore standard enthalpy and entropy of reaction Eq. (1) for YCoO_3 may be roughly estimated by averaging corresponding standard enthalpies and entropies for Er- and Ho-containing cobaltites.

The thermodynamic quantities of reaction Eq. (1) obtained in this way are also

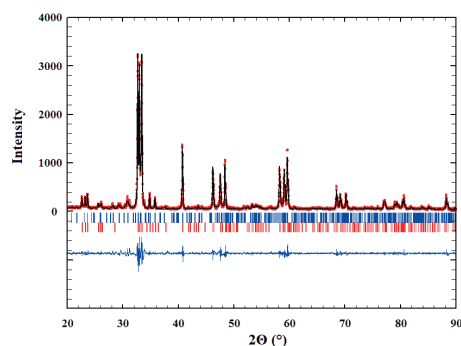


Fig. 5. X-ray diffraction pattern and its matching refinement plot of $\text{YBaCo}_2\text{O}_{6-\delta}$ obtained by annealing at 1000°C in oxygen for 72 h: observed X-ray diffraction intensity – points and calculated curve – line. The bottom curve is the difference of patterns, $y_{\text{obs}} - y_{\text{cal}}$, and the small bars indicate the angular positions of the allowed Bragg reflections for $\text{YBaCo}_2\text{O}_{6-\delta}$ with $3\times 2\times 2$ superstructure (blue) and $1\times 2\times 2$ superstructure (red)

shown in Table 2. They allow estimating corresponding equilibrium decomposition temperatures for YCoO_3 in air and oxygen. As seen in Table 2 this estimation gives 953 °C as the decomposition temperature of YCoO_3 in air, which is in line with that reported earlier [11–13, 22, 23]. The value of decomposition temperature in oxygen is around of 1060 °C. Taking into account that this is only a very rough estimation one may expect the real decomposition temperature for YCoO_3 in oxygen in the range of 1000–1100 °C, i. e. during annealing of the $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ equimolar mixture at 1000 °C

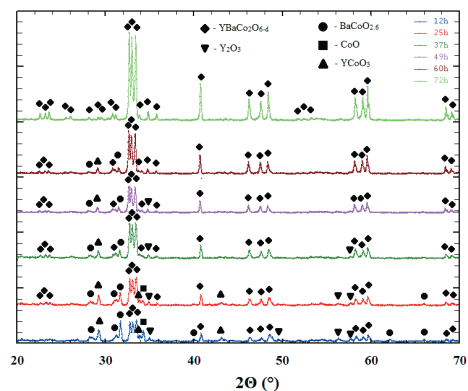


Fig. 6. XRD patterns of $\text{YCoO}_{3-\delta}$ and $\text{BaCoO}_{3-\delta}$ equimolar mixture step-by-step annealed at 1000 °C in pure oxygen

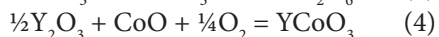
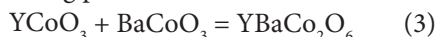
Table 2

Thermodynamics of reaction Eq. (1) for the selected cobaltites at 927 °C					
	ΔH° , kJ·mol ⁻¹	ΔS° , J·mol ⁻¹ ·K ⁻¹	T_{air}^* , °C	$T_{\text{O}_2}^{**}$, °C	Reference
HoCoO_3	44.88	30.63	1051	1192	[24, 25]
ErCoO_3	51.34	41.3	866	970	[24, 25]
YCoO_3	48	36	953	1060	Estimated in this work

* Equilibrium temperature for RCoO_3 (R = Y, Ho, Er) decomposition in air

** Equilibrium temperature for RCoO_3 (R = Y, Ho, Er) decomposition in oxygen

in pure oxygen atmosphere its first component is in equilibrium with oxides Y_2O_3 and CoO . Therefore synthesis of the $\text{YBaCo}_2\text{O}_{6-\delta}$ double perovskite under these conditions can be described by the following parallel reactions



The equilibrium of reaction Eq. (4) is shifted to the right due to consumption of YCoO_3 as a reagent of reaction Eq. (3).

Comparison of the results of synthesis at 1000 °C in two atmospheres, i. e. air and oxygen, shows that in the second case formation of the double perovskite occurs apparently faster. One may speculate on the reasons of the observed positive influence of high oxygen pressure. Intuitively

it seems quite expected that the combination (or interaction) of two ‘simple’ perovskites representing elementary ‘building’ units of the double perovskite structure is a faster process than a combination of barium cobaltite with two oxides. Significant diffusion difficulties are quite expected in the last case. However the exact reasons and detailed microscopic mechanism of an interaction in oxygen or air atmosphere should be studied in order to make meaningful conclusions. We only would like to emphasize once again the key role, which thermodynamic stability of YCoO_3 plays in the optimization of synthesis routine for the $\text{YBaCo}_2\text{O}_{6-\delta}$ double perovskite.

Conclusions

Synthesis of $\text{YBaCo}_2\text{O}_{6-\delta}$ from equimolar mixture of YCoO_3 and $\text{BaCoO}_{3-\delta}$ was studied at 900 °C and 1000 °C in air and pure oxygen atmosphere. It was shown that synthesis at 1000 °C in pure oxygen atmosphere is an optimal way of obtaining the single phase $\text{YBaCo}_2\text{O}_{6-\delta}$. Detailed step-by-step investigation of the synthe-

sis was carried out at 1000 °C in $p\text{O}_2 = 1$ atm. The mechanism of $\text{YBaCo}_2\text{O}_{6-\delta}$ synthesis in different gas atmospheres was proposed based on thermodynamics of YCoO_3 and crucial role of this oxide stability in governing of the synthesis process was revealed.

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