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RESEARCH ARTICLE

Alcoxotechnology for obtaining heat-resistant materials based on rhenium and ruthenium

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Objectives. *To develop physical and chemical bases and methods to obtain rhenium–ruthenium isoproxide $Re_{4-y}Ru_yO_6(OPr^i)_{10}$—a precursor for obtaining a high-temperature alloy—from ruthenium acetylacetonate and rhenium isoproxide acquired by electrochemical methods.*

Methods. *IR spectroscopy (EQUINOX 55 Bruker, Germany), X-ray phase and elemental analyses, energy-dispersive microanalysis (EDMA, SEM JSM5910-LV, analytical system AZTEC), powder X-ray diffraction (diffractometer D8 Advance Bruker, Germany), experimental station XSA beamline at the Kurchatov Synchrotron Radiation Source.*

Results. *The isoproxide complex of rhenium–ruthenium $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ was obtained, and its composition and structure were established. Previously conducted quantum chemical calculations on the possibility of replacing rhenium atoms with ruthenium atoms in the isopropylate complex were experimentally proven, and the influence of the electroconductive additive on the composition of the obtained alloy was revealed.*

Conclusions. *Physical and chemical bases and methods for obtaining rhenium–ruthenium isoproxide $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ were developed. The possibility of using rhenium–ruthenium $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ as a precursor in the production of ultra- and nanodisperse rhenium–ruthenium alloy powders at a record low temperature of 650°C were shown.*

Keywords: *alkoxotechnology, electrochemical synthesis, rhenium isopropoxide, ruthenium acetylacetonate, rhenium–ruthenium isopropoxide, rhenium–ruthenium alloy, low-temperature reduction*

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НАУЧНАЯ СТАТЬЯ

Алкоксотехнология получения жаропрочных материалов на основе рения и рутения

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Цели. *Разработка физико-химических основ и способов получения изопроксида рения-рутения $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ из ацетилацетоната рутения и изопроксида рения, полученного электрохимическим методом – прекурсора получения высокотемпературного сплава.*
Методы. *ИК-спектроскопия (EQUINOX 55 Bruker, Германия), рентгенофазовый и элементный анализ, энергодисперсионный микроанализ (ЭДМА, СЭМ JSM5910–LV, аналитическая система AZTEC), порошковая рентгеновская дифракция (дифрактометр D8 Advance Bruker, Германия), экспериментальная станция «РСА» Курчатовского источника синхротронного излучения.*
Результаты. *Получен изопроксидный комплекс рения-рутения $Re_{4-y}Ru_yO_6(OPr^i)_{10}$, подтверждены его состав и строение. Экспериментально подтверждены ранее проведенные квантово-химические расчеты, свидетельствующие о возможности замещения атомов рения атомами рутения в изопропилатном комплексе. Выявлено влияние электропроводящей добавки на состав получаемого сплава.*
Выводы. *Разработаны физико-химические основы и предложены способы получения изопроксида рения-рутения $Re_{4-y}Ru_yO_6(OPr^i)_{10}$, который может найти применение в качестве предшественника при получении ультра- и нанодисперсных порошков сплавов рений-рутений при рекордно низкой температуре 650°C.*

Ключевые слова: *алкоксотехнология, электрохимический синтез, изопроксид рения, ацетилацетонат рутения, изопроксид рения-рутения, сплав рения-рутения, низкотемпературное восстановление*

Для цитирования: Куликова Е.С., Чернышова О.В., Носикова Л.А., Светогоров Р.Д., Дробот Д.В., Михеев И.А. Алкоксотехнология получения жаропрочных материалов на основе рения и рутения. *Тонкие химические технологии.* 2020;15(6):67-76. https://doi.org/10.32362/2410-6593-2020-15-6-67-76

INTRODUCTION

Functional materials based on rhenium have a variety of applications, such as in aerospace and power engineering as well as in the production of catalysts for processing renewable and nontraditional raw materials [1, 2]. Alloys containing rhenium are resistant to high temperatures and aggressive environments and have high strength performance and wear resistance.

The classic methods for producing rhenium-containing alloys, such as induction and electric arc melting and powder metallurgy, have significant disadvantages: high process temperatures (over 2500°C), the inability to uniformly introduce alloying components or control the microstructure of samples, and enormous energy costs [1, 2]. Thus, in the last two decades, vacuum smelting in induction and arc furnaces with consumable electrodes, directional crystallization, and heat treatments have been widely used [3–5].

Alcoxotechnology is a promising method for obtaining alloy powder precursors with a given set of properties and allows for obtaining ultrafine and nanoscale materials of high phase and chemical purity at low temperatures (below 950°C).

Oxoalcoxy derivatives of rhenium can act as such precursors. Rhenium isoproxide is of particular interest as rhenium atoms form an almost regular rhombus in its structure [6, 7].

Using quantum chemical calculations in the Priroda 06 program, the possibility of replacing rhenium atoms with ruthenium atoms in an isoproxide complex while preserving the tetranuclear cluster has been shown [8]. Ruthenium is chosen because in the periodic table, it is in close proximity to rhenium, and both elements are transients and have close atomic radii values. Moreover, it has been known that the addition of ruthenium increases the temperature limit for heat-resistant nickel alloys [9, 10].

The aim of this study is to develop physical and chemical bases and methods for obtaining rhenium–ruthenium isoproxide $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ to establish its composition, structure, and use as a precursor in the preparation of ultra- and nanodisperse powders of rhenium–ruthenium alloys.

EXPERIMENTAL

The synthesis of the rhenium–ruthenium isoproxide complex $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ was performed in three stages: synthesis of rhenium isoproxide, synthesis of ruthenium acetylacetonate, and direct synthesis of the rhenium–ruthenium isoproxide complex.

Rhenium isoproxide was synthesized by an electrochemical method according to the methods in [11, 12]. For the synthesis, a cell without a separated anode and cathode space with a thermostatic jacket was used. Platinum was used as the cathode, and rhenium was used as the anode. The voltage was 130–210 V, the current was 30–80 mA, and the alcohol volume was 200 mL. The synthesis time was 92 h in the presence of a background electrolyte of lithium chloride (C_{LiCl} = 0.1 mol/L) and 28 h in the presence of tetrabutylammonium bromide ($C_{C16H36BrN}$ = 2.5% by weight of alcohol). At the stage of obtaining the rhenium isoproxide, the following reagents were used: metal plates of rhenium and platinum (purity 99.9%), metal sodium (purity 99.9%), isopropyl alcohol (TU 2632-011-29483781-2009, extra-pure grade), lithium aluminum hydride (*Merck KgaA*, pure), lithium chloride (*Merck KgaA*, pure), and tetra-butylammonium bromide (TU 6-09-1859-77, pure). Isoproxides are sensitive to moisture, so the isopropyl alcohol was dried by sequentially introducing sodium into the system at the first stage and lithium aluminum hydride at the second stage when heated. The final water content was less than 0.01%. The lithium chloride was dehydrated according to the method described in [13]. Similarly, the tetrabutylammonium bromide was dehydrated.

Ruthenium (III) acetylacetonate was obtained by the method described in [14]. The following reagents were used in the synthesis: acetylacetone (*Merck KgaA*, purity ≥ 99%, chemically pure), acetone (TU 6-09-3513-86, extra-pure grade), ruthenium hydrochloid (purity 41.67%), sodium bicarbonate (GOST 4201-79, chemically pure), ethanol (*Merck KgaA*, purity ≥ 99%, pure). The product yield was 60%, and the obtained ruthenium acetylacetonate crystals were studied by X-ray phase analysis.

The following method for the rhenium–ruthenium isoproxide synthesis was proposed. The synthesis was carried out by mixing rhenium isoproxide and ruthenium acetylacetonate in a conical flask in a "dry" box. The ratio of rhenium to ruthenium 1 : 1 was chosen from quantum chemical calculations [8]. When the ruthenium acetylacetonate was added, the rhenium isoproxide solution changed in color from brownish-green to blood red. Next, the solution was heated to boiling in a reflux flask for 3 h with constant stirring. The resulting complex was identified by IR spectroscopy and elemental analysis.

The resulting alkoxy rhenium complex was used as a precursor in the production of the rhenium–ruthenium alloy powders. Two methods were used to obtain it. In the first method, pre-evaporated rhenium–ruthenium isoproxide in a porcelain boat was placed in a recovery unit (flow reactor), after which all connections of the unit were sealed. Before starting the synthesis, the system was purged with an inert gas (argon) for 30 min. After the purging, hydrogen was supplied, and recovery was performed for 60 min under the following conditions: hydrogen flow, 4 L/h; pressure, 1 atm. The temperature conditions in the first experiments were set in the range of 400–450°C but, in subsequent experiments, were adjusted to 600–650°C. After the recovery process was completed, an inert atmosphere was created in the system for 30 min. Then, the furnace was turned off, and after it was cooled, the sample boats were removed and weighed.

The flow reactor for the reduction of the rhenium–ruthenium isoproxide could not provide a hydrogen pressure of more than 1 atm, so the second method for producing alloy powders in a hydrogen atmosphere using an autoclave installation was used. Evaporated and weighed samples in porcelain boats were immersed in the autoclave. Before the recovery process was started, the system was purged with hydrogen, creating an excess of it in the installation. After 10 min of purging, the hydrogen supply was turned off, and the final pressure was set to 5 atm. Next, the furnace heating was connected, and the rhenium–ruthenium isoproxide reduction was performed at 650°C for 90 min, after which the cooled samples were removed and reweighed. The autoclave method was used to obtain samples from precursors that did not contain lithium chloride. Samples obtained by the two methods described were examined using X-ray phase analysis.

The infrared spectra of liquids in the region of 3600–50 cm^{-1} were recorded on the EQUINOX 55 Bruker (Germany) device [15, 16]. The resolution of the device was 2 cm^{-1}, and the accuracy of the wavenumber determination was 0.1 cm^{-1}. Energy-dispersive microanalysis was used to analyze the distribution of elements in the samples (SEM JSM5910LV, AZTEC analytical system).

The powder X-ray diffraction examinations were performed with two installations:

1. The D8 Advance Bruker diffractometer, Germany (Cu Kα radiation, sample rotation, continuous [1 deg/min], step-by-step [0.02° step, 10 s exposure] modes in the angle range 2Θ = 5°–100°) [17]. The ICDD PDF-2 and Crystallography Open Database[1] were used for phase identification.

2. The XSA experimental station [18] of the Kurchatov Synchrotron Radiation Source. To measure the diffraction patterns, we used monochromatic radiation with wavelength λ = 0.802575 Å (photon energy E = 15448.3 eV) focused on a 400 × 400-μm sample. The survey was carried out in the Debye–Scherrer geometry (for transmission) using a two-dimensional Rayonix SX165 detector located perpendicular to the synchronous radiation beam at a distance of 80 mm. The sample was placed in a cryoloop with a diameter of 300 μm and rotated around the horizontal axis during the measurement to average the diffractograms over the sample orientations. The exposure time was 3–5 min. The two-dimensional patterns obtained at the detector were integrated, i.e., reduced to the standard one-dimensional form of the intensity dependence on the scattering angle I (2Θ) in the Dionis program [19]. The angular scale of the detector was calibrated, and the hardware broadening of the diffraction reflexes was determined by measuring polycrystalline standard LaB_6 (NIST SRM 660a). The qualitative phase composition was determined in the Match! program[2] using the PDF-4+ powder database based on the corundum number method [20].

RESULTS AND DISCUSSION

The rhenium isoproxides obtained by the anodic dissolution of rhenium in isopropyl alcohol were studied by a set of physical and chemical methods.

All studied solutions had identical absorption bands in the IR spectra regardless of the selected conductive additive: 3350–3310 cm^{-1}, ν(O–H); 2970–2928 cm^{-1}, $\nu_{str}(C–H)_{(as)}$; 2885–2830 cm^{-1}, $\nu_{str}(C–H)_{(s)}$; 1466–1456 cm^{-1}, $\nu_{as}(C–C)$; 1376–1306 cm^{-1}, ν(C–C); 1161–1128 cm^{-1}, ν(C–O); 950–906 cm^{-1}, ν(Re=O); 817–815 cm^{-1}, ν(Re–O–(R)–Re) (bridge); 678–602 cm^{-1}, ν(Re–O–(R)); 486–463 cm^{-1}, ν(Re–O–(R)); 427–414 cm^{-1}, $\nu_a(Re–O)$; 356–318 cm^{-1}, $\nu_s(Re–O)$; 287–201 cm^{-1}, ν(Re–Re); 213–166 cm^{-1}, δ(O–Re–O) + δ(O–Re); 137–104 cm^{-1}, δ(O–Re–O); and 92 cm^{-1}, t(Re–Re) [21–25].

The elemental composition of the pre-evaporated samples was examined. Figure 1 shows electron microscopy images of rhenium isoproxide, displaying the areas for which the elemental analysis was performed (Table 1).

The small amount of copper impurities was due to the fact that during the electrochemical synthesis, copper wire was used as a current supply

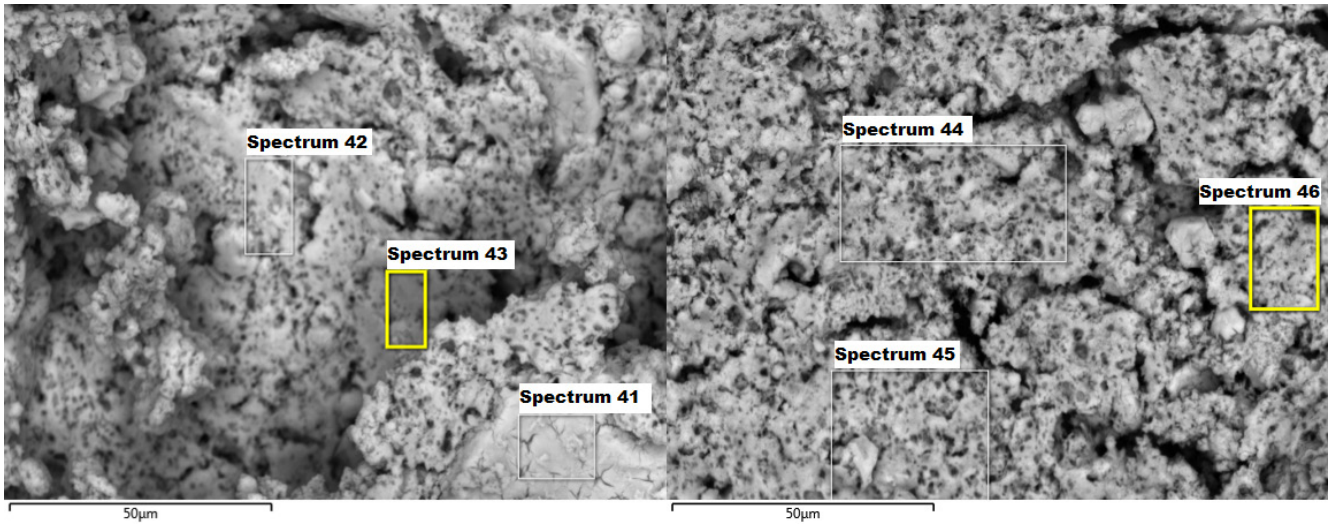

Fig. 1. Electron microscopy of rhenium isoproxide.

[1] http://www.crystallography.net/cod/. Accessed May 15, 2020.
[2] Match! – Phase Analysis using Powder Diffraction, Crystal Impact – Dr. H. Putz & Dr. K. Brandenburg GbR, Kreuzherrenstr. 102, 53227 Bonn, Germany, https://www.crystalimpact.de/match. Accessed September 03, 2020.

to the rhenium anode. The presence of aluminum was because $Li[AlH_4]$ was used for dewatering the isopropyl alcohol, and the unreacted aluminum remained in the solution.

The synthesized ruthenium acetylacetonate was studied using X-ray phase analysis on two installations (D8 Advance Bruker, Germany, and XSA diffractometer, Belok station, Russia), which showed that the product was single-phase with the cell parameters indicated in Table 2.

The obtained $Ru(AcAc)_3$ was characterized by elemental analysis (for the C, H, N, and S content). In the $Ru(AcAc)_3$ (m = 2.995 mg), the carbon content was 51.41%, and the hydrogen content was 6.44%, corresponding to the theoretical calculations. Nitrogen and sulfur were not detected.

The IR spectra in the range of 4000–50 cm^{-1} were studied for the obtained rhenium–ruthenium isoproxides. In the investigated range, the following absorption bands are observed: 3350–3310 cm^{-1}, ν(O–H); 2970–2928 cm^{-1}, $\nu_{str}(C–H)_{(as)}$; 2885–2830 cm^{-1}, $\nu_{str}(C–H)_{(s)}$; 1466–1456 cm^{-1}, $\nu_{as}(C–C)$; 1376–1306 cm^{-1}, ν(C–C); 1120–1114 cm^{-1}, ν(C–O); 949–910 cm^{-1}, ν(Re=O); 817–816 cm^{-1}, ν(Re–O–(R)–Re) (bridge); 678–612 cm^{-1}, ν(Re–O–(R)); 583–520 см$^{-1}$, ν(Ru–C); 493–453 cm^{-1}, ν(Re–O–(R)); 440–410 cm^{-1}, ν(C–Ru–C); 385–344 cm^{-1}, ν(Ru–C–O); 325–301 cm^{-1}, ν(Re–O); 286–229 cm^{-1}, ν(Re–Re); 212–162 cm^{-1}, δ(O–Re–O) + + δ(O–Re); 159–144 cm^{-1}, ν(Ru–Ru); 140–111 cm^{-1}, ν(Ru–Re); 107–88 cm^{-1}, ν((CO)–Ru–(CO)); 92 cm^{-1}, t(Re–Re) и 89–80 cm^{-1}, ν(C–Ru–C); and 76–59 cm^{-1}, $\nu_{dis}(RuCO_3)$.

The absorption bands Re–Ru, Re–Re, and Ru–Ru were present in the 950–59-cm^{-1} spectral region, which confirmed the presence of bonds as established by quantum chemical calculations and experimentally proved the possibility of replacing rhenium atoms with ruthenium atoms in the rhenium–ruthenium isoproxide complex.

The elemental composition of the surface was studied for the pre-evaporated rhenium–ruthenium isoproxide samples. Figure 2 shows electron microscopy images of the rhenium–ruthenium isoproxide; the selected regions are the points for which elemental analysis was performed (Table 3).

The presence of copper and aluminum impurities in the samples was explained in the same way as for the rhenium isoproxide samples.

The products obtained during the hydrogen reduction process were characterized by X-ray

Table 1. Elemental analysis of rhenium isoproxide

Spectrum	C	O	Al	Cu	Re	Sum of atoms, %
41	62.24	27.86	–	–	9.90	100
42	59.06	19.74	0.40	–	20.80	100
43	61.08	25.66	–	–	13.26	100
44	69.70	21.64	–	0.14	8.52	100
45	67.53	24.09	–	0.14	8.24	100
46	67.48	25.45	–	0.11	6.96	100

Table 2. Comparative table of the X-ray diffraction data of ruthenium acetylacetonate

Cell parameters, Å		
D8 Advance Bruker	Diffractometer of the Belok station	Data PDF-4+ Organic, No. 02-066-6916 monoclinic structure, gr. P21/c, cell parameters: a = 14.025, b = 7.533, c = 16.309, β = 99°
a = 14.040, b = 7.552, c = 16.364, β = 98.93°	a = 14.024, b = 7.530, c = 16.376, β = 98.90°	

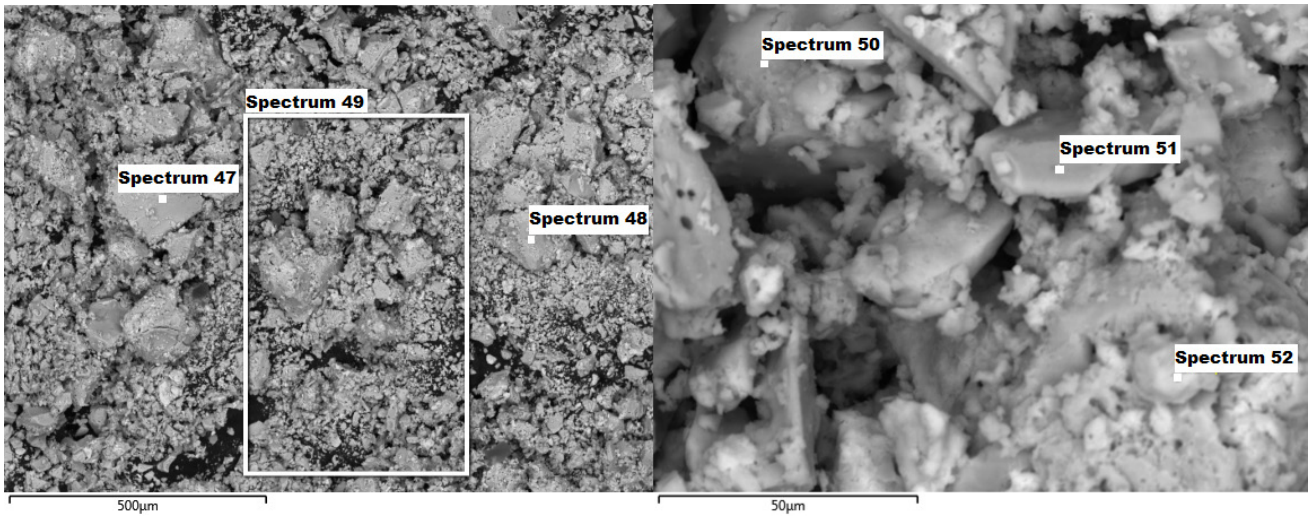

Fig. 2. Electron microscopy of rhenium–ruthenium isoproxide.

Table 3. Elemental analysis of rhenium–ruthenium isoproxide

Spectrum	C	O	Al	Cu	Ru	Re	Sum of atoms, %
47	62.67	27.44	2.40	–	2.00	5.49	100
48	58.96	19.89	2.37	–	2.80	15.98	100
49	61.39	24.43	–	0.42	2.05	11.71	100
50	69.23	22.03	–	0.17	2.94	5.63	100
51	67.83	23.89	2.39	–	3.07	2.82	100
52	67.02	25.95	–	–	–	7.03	100

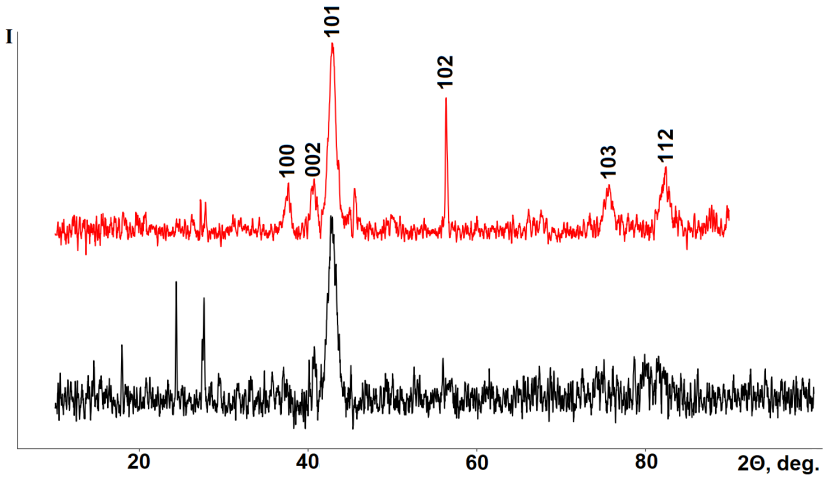

Fig. 3. Results of X-ray phase analysis of the rhenium–ruthenium alloy
(black is the sample obtained from the flow reactor, and red is the sample obtained from the autoclave).

Table 4. Comparative table of the radiometric data of the rhenium–ruthenium alloy

Synthesis	Cell parameters, Å		
	D8 Advance Bruker	Diffractometer of the Belok station	PDF-2, PDF-4+, COD databases Hexagonal ($P6_3/mmc$)–$Re_{0.9}Ru_{0.1}$
In a flow reactor	$Re_{0.9}Ru_{0.1}$ a = 2.771, c = 4.424	$Re_{0.9}Ru_{0.1}$ a = 2.779, c = 4.439	$Re_{0.9}Ru_{0.1}$ a = 2.753, c = 4.432
In an autoclave	$Re_{0.9}Ru_{0.1}$ a = 2.757, c = 4.430	$Re_{0.9}Ru_{0.1}$ a = 2.754, c = 4.424	

phase analysis. The powder samples obtained from the rhenium–ruthenium isoproxide at 400–450°C were X-ray amorphous. When the temperature was increased to 650°C, the obtained rhenium–ruthenium samples were double-phase. The main phase was $Re_{0.9}Ru_{0.1}$. The cell parameters are shown in Table 4. The impurity phase was $LiReO_4 \cdot H_2O$ with the following parameters: a = 5.712 Å, b = 10.782 Å, c = 7.453 Å, β = 102.49° (card number 52-1579, P21/a, cell parameters: a = 5.671 Å, b = 10.770 Å, c = 7.475 Å, β = 102.45°). The samples obtained by the autoclave method at the same temperature were single-phase and of the composition $Re_{0.9}Ru_{0.1}$ (Fig. 3, Table 4).

The phase composition was refined using powder X-ray diffraction on a synchrotron radiation source. Comparative parameters are given in Table 4.

Based on the results of the X-ray phase analysis and the rhenium–ruthenium phase diagram [26], it was concluded that the samples obtained were rhenium–ruthenium alloys.

The sizes of the alloy particles calculated from the width of the diffraction peaks were 7.2 nm according to the Scherrer formula and 16.5 nm according to the Williamson–Hall method, which confirms the receipt of nanoscale samples.

CONCLUSIONS

In this study, a method for obtaining a rhenium–ruthenium isoproxide complex from rhenium isopropylate $Re_4O_6(OPr^i)_{10}$, and ruthenium acetylacetonate $Ru(AcAc)_3$ was developed. The resulting complex was characterized by a set of methods that confirmed previously performed quantum chemical calculations. It was shown that the $Re_{4-y}Ru_yO_6(OPr^i)_{10}$ complex formed nanodisperse powders of rhenium–ruthenium alloys at low-temperature reduction (650°C) in a hydrogen atmosphere. Moreover, the effect of an electrically conductive additive on the resulting alloy's composition was demonstrated. The use of lithium chloride led to the formation of the $LiReO_4$ phase in the final product in contrast with tetrabutylammonium bromide, which was not involved in the formation of the intermediate phases in the rhenium–ruthenium alloy.

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Authors' contribution

E.S. Kulikova – conducting syntheses of research, review of publications on the topic of the article, collection and processing the material, writing the text of the article;

O.V. Chernyshova – consultation for carrying out of separate stages of research, scientific editing;

L.A. Novikova – conducting research and consultation on processing the obtained results;

R.D. Svetogorov – conducting research using powder X-ray diffraction on the synchrotron radiation source, processing the obtained results;

D.V. Drobot – development of the concept of scientific work, critical revision with the introduction of valuable intellectual content, consultation on all stages of research;

I.A. Mikheev – collection and processing the material, statistical processing.

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