

Synthesis of nanosized pure and Cr doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ solid solutions

A. S. Yordanova^{1*}, R. S. Iordanova¹, V. S. Nikolov¹, I. I. Koseva¹, P. T. Tzvetkov¹

¹ Institute of General and Inorganic Chemistry, Bulgarian Academy of Sciences,
Acad. G. Bonchev Str., Building 11, 1113 Sofia, Bulgaria

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Nanosized solid solutions with the formula $\text{Sc}_{2-x-y}\text{In}_x\text{Cr}_y(\text{WO}_4)_3$, where x varies from 0 to 2 and y from 0.02 to 0.06 were synthesized for the first time by co-precipitation method. Phase and structural investigations were performed. By X-Ray analysis it was established that only by strict maintenance of pH between 2.7 and 5.7 a pure, monophasic product can be obtained. It was found that at room temperature the solid solutions $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ at x value equal to 0 and 0.5 are orthorhombic and for x value between 1.0 and 2.0 are monoclinic. The lattice parameters of the obtained solid solutions were calculated. The conditions of thermal treatment of the amorphous precipitates were determined by preliminary DTA and TG analyses. The average crystallite dimensions of the obtained solid solutions, calculated from the X-ray data, are between 19 and 110 nm. The crystallite sizes depends on the ratio of Sc, In and Cr, and on the temperature of thermal treatment.

Keywords: Nanostructured materials, Tungstates, X-ray diffraction, DTA.

INTRODUCTION

Scandium tungstate $\text{Sc}_2(\text{WO}_4)_3$ and indium tungstate $\text{In}_2(\text{WO}_4)_3$ belong to a class of compounds with a general formula $\text{Me}_2(\text{WO}_4)_3$, where $\text{Me} = \text{Y}, \text{Sc}, \text{In}, \text{Al}$ or lanthanides with a small ionic radius – Ho, Er, Tm, Yb, Lu. Normally, these compounds crystallize in an orthorhombic structure, space group Pnca [1]. As a result of phase transition, the structure transforms to monoclinic at lower temperatures, with space group $\text{P2}_1/\text{a}$. The phase transition of $\text{In}_2(\text{WO}_4)_3$ occurs at 258 °C [2], and of $\text{Sc}_2(\text{WO}_4)_3$ occurs at minus 263 °C [3].

The specificities of the orthorhombic structure, Pnca space group, determine a number of interesting properties and potential applications of the considered class of compounds. The orthorhombic modification is built of MeO_6 octahedrons, connected with WO_4 tetrahedrons in such a manner that they form a layered structure with a large tunnel size, where the Me^{3+} ions are sufficiently mobile, i.e. the compounds possess Me^{3+} ionic conductivity. The trivalent ionic conductivity of $\text{Al}^{3+}, \text{Sc}^{3+}, \text{Y}^{3+}, \text{Er}^{3+}$ and In^{3+} in the corresponding tungstates is directly demonstrated and described in reference literature [4–7].

The increase of the lattice parameters by changes in the chemical composition would result in the increase of the ionic conductivity too. Similar behaviour has been already demonstrated in the system $\text{Al}_2(\text{WO}_4)_3 - \text{Sc}_2(\text{WO}_4)_3 - \text{Lu}_2(\text{WO}_4)_3$ [8–10].

Some of the $\text{Me}_2(\text{WO}_4)_3$ compounds possess unusually low thermal expansion coefficients, including zero and even negative ones, within a broad temperature range [11–14]. For example, a zero thermal expansion coefficient is reported for the $\text{Al}_{1.68}\text{Sc}_{0.02}\text{In}_{0.3}(\text{WO}_4)_3$ composition [15–17].

Another basic application of this class of compounds is as laser media for tunable lasers. The Me^{3+} situated in octahedral coordination may be easily substituted by a Cr^{3+} active ion, characterized by a broad absorption cross section, as well as by a broad emission, being incorporated in a structure with weak or intermediate type crystal field [18–20]. Our previous investigations on optical and phonon properties of nanocrystalline Cr doped $\text{Al}_2(\text{WO}_4)_3$ and solid solutions of $\text{Al}_{2-x}\text{Sc}_x(\text{WO}_4)_3$ revealed the presence of two chromium sites in these structures, one with intermediate type and another with weak crystal field strength, respectively. [21, 22].

An important advantage of this class of compounds is their aptitude to build their structure by accumulation of different trivalent ions. This provides the possibility to tailor the thermal expansion coefficient, ionic conductivity and laser emission to a desired value by varying the chemical composition.

* To whom all correspondence should be sent:
E-mail: a.yordanova@svr.igic.bas.bg

tion of solid solutions within the mentioned structures.

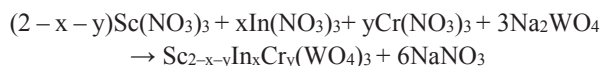
For the solid solutions with composition $Sc_{2-x}In_x(WO_4)_3$, pure or doped with chromium there is no data in the reference literature. This refers to both the methods for their production (as a rule using only the classical solid state synthesis) and the already discussed properties, important for their application.

This article presents the results of the synthesis of nanosized $Sc_{2-x}In_x(WO_4)_3$ solid solutions in the entire concentration range (for x values from 0 to 2), as well as the same solutions doped with 1 and 3 at.% Cr by co-precipitation method.

EXPERIMENTAL

The nanosized powders from solid solutions with a general formula $Sc_{2-x}In_x(WO_4)_3$ for x value equal to 0, 0.5, 1.0, 1.5 and 2 were obtained by co-precipitation method. In addition to the pure solid solutions, two series of solid solutions doped by Cr^{3+} with the same atomic ratio of Sc to In were also synthesized. These series of Cr^{3+} doped solid solutions was with general formula $Sc_{2-x-y}In_xCr_y(WO_4)_3$ for values of y = 0.02 and 0.06. In other words, the obtained solid solutions were with concentration of In 0, 25, 50, 75 and 100 at.% (100, 75, 50, 25 and 0 at.% Sc respectively) and with concentration 0, 1 and 3 at.% of Cr^{3+} with respect to the sum of Sc and In. Each of the solid solutions was synthesized using two preliminarily prepared separate aqueous solutions: first solution of Na_2WO_4 in 50 ml of distilled water using $Na_2WO_4 \cdot 2H_2O$ (p.a.) as a reagent, and second solution of $Sc(NO_3)_3$, $In(NO_3)_3$ and $Cr(NO_3)_3$ in 50 ml of distilled water using $Sc(NO_3)_3 \cdot 4H_2O$, $In(NO_3)_3 \cdot 3H_2O$ and $Cr(NO_3)_3 \cdot 9H_2O$ (p.a.) as reagents. The quantity of the tungstates in the first solution and the total amount of nitrates in the second one corresponded to the stoichiometric composition for the progress of the co-precipitation reaction:

Mixing:



The two aqueous solutions were homogenized at room temperature until full clarification. The prepared solutions were mixed by simultaneous pouring in a third glass vessel and the obtained white substance was intensively mixed in the course of 1 hour. The solutions were adjusted to the desired pH value by adding diluted nitric acid (1:3), (HNO_3 p.a., 63.01 g/mol), or 1M sodium hydroxide (p.a.). The precipitation process was in the course of 24 hours.

The obtained precipitate was filtered, dried at 80 °C, then ground in an agate mortar, washed with deionised water and dried again. The amorphous precipitates of the solid solution were thermally treated in two single series: at 550 °C, 650 °C, 700 °C, 800 °C for 1 hour and at 900 °C for 3 hours. After treatment at 550 °C, 650 °C, 700 °C and 800 °C, the obtained powders were used to determine the crystallite sizes depending on the composition of the solid solutions and the temperature of thermal treatment. The well crystallized powders at 900 °C for 3 hours were used to determine structure, cell parameters and lattice volume depending on the composition of the solutions.

Structural characterization was carried out by powder X-ray diffraction (XRD) using a Bruker D8 Advance powder diffractometer with Cu K α radiation and SolX detector. XRD spectra were recorded at room temperature. Data were collected in the 2 θ range from 10 to 80 deg with a step 0.04 deg and 1s/step counting time. XRD spectra were identified using the Diffractplus EVA program. The obtained data were used for determining of the solid solution lattice parameters. The mean crystallite size and unit cell parameters were calculated from the integral breadth of all peaks (Pawley fit) using the TOPAS 3 program.3 – General profile and structure analysis software for powder diffraction data, 2005, Bruker AXS, Karlsruhe, Germany.

The thermal analysis was used for determination of crystallization temperature of some solid solution precipitate as well as for establishing of the phase transition temperature. The investigations were done by combined LABSYS™ EVO DTA/TG device of the SETARAM Company, France. The samples were investigated at a heating rate of 10°C/min in Ar flow at a flow rate of 20 ml/min.

RESULTS AND DISCUSSION

Our earlier investigations on synthesizing nanosized $Al_{2-x}In_x(WO_4)_3$ [23] and $Al_{2-x}Sc_x(WO_4)_3$ [24] by the co-precipitation method proved that aluminium – scandium solid solutions can be obtained without any special adjustment of the pH value of the mixed solution. Pure $Sc_2(WO_4)_3$ is obtained at pH equal at 5.7. In contrast for $Al_{2-x}In_x(WO_4)_3$, it was necessary to adhere to a strict pH value of the solution. A pure $In_2(WO_4)_3$ product was obtained only for pH values between 2.7 and 2.9. A two-phase product ($In_2(WO_4)_3$ and In_6WO_{12} or $In_2(WO_4)_3$ and WO_3) was obtained for the other pH values [23]. Therefore it was logical to presume that for obtaining pure $Sc_{2-x}In_x(WO_4)_3$ solid solutions, it is necessary to adjust intermediate pH values between 5.7 and 2.8, proportionally diminishing with increasing

the concentration of indium in the solid solution. In this way, in order to obtain solid solutions of $Sc_{2-x}In_x(WO_4)_3$ with x equal to 0.5, 1.0, and 1.5, the chosen pH values of the solutions were 4.97, 4.25 and 3.53 respectively. The solutions were adjusted to the desired pH value by adding diluted nitric acid or sodium hydroxide.

Well crystallized samples of $Sc_{2-x}In_x(WO_4)_3$ were obtained after thermal treatment of the precipitates at 900 °C for 3 h. (t. 1). The X-ray pattern of the products for values of x equal to 2.0, 1.5 and 1.0 corresponds exactly to the reference literature data for the monoclinic symmetry with space group $P2_1/a$ and x values equal to 0.5 and 0 correspond to the orthorhombic symmetry (space group $Pnca$) [2, 3]. As pointed out in the literature data for $In_2(WO_4)_3$, the two structures are close to each other and in the X-ray pattern of monoclinic symmetry two additional peaks at $2\theta = 23.6$ and 25.7 are observed. These peaks are marked by asterisk on the Figure 1b. There are not any additional peaks of impurities in the obtained solid solutions. The missing peaks of an admixture phase in the X-ray patterns and the shift of peaks at higher values 2θ with the increasing of the In content is a proof that pure sol-

id solution compounds were obtained in the entire range from $Sc_2(WO_4)_3$ to $In_2(WO_4)_3$. The calculated lattice parameters of the pure $Sc_{2-x}In_x(WO_4)_3$ and Cr doped $Sc_{2-x-y}In_xCr_y(WO_4)_3$ (6–9) solid solutions are shown in Table 1.

As can be seen from Table 1, lattice parameters and cell volume of the solutions with orthorhombic symmetry slightly increase (solid solutions number 1,2 and 6,7) with addition of indium. These results are expected due to the small difference in ionic radii of scandium (0.745 Å) and indium (0.8 Å). On the other hand due to a big difference between the ionic radii of scandium (indium) and chromium (0.615 Å) ions, a clear tendency of lattice parameters decreasing was observed with the addition of Cr^{3+} in the orthorhombic (1, 2 and 6, 7) and monoclinic structure (3÷5 and 8÷10). The observed slight non – regular lattice parameters deviation of the of solid solutions with monoclinic symmetry is probably a result of the different sites Cr^{3+} ions (scandium or indium sites) could occupy into the solid solutions.

The aim of the second series of thermal treatment of the precipitates was to obtain nanosized powders from these solid solutions. The amorphous

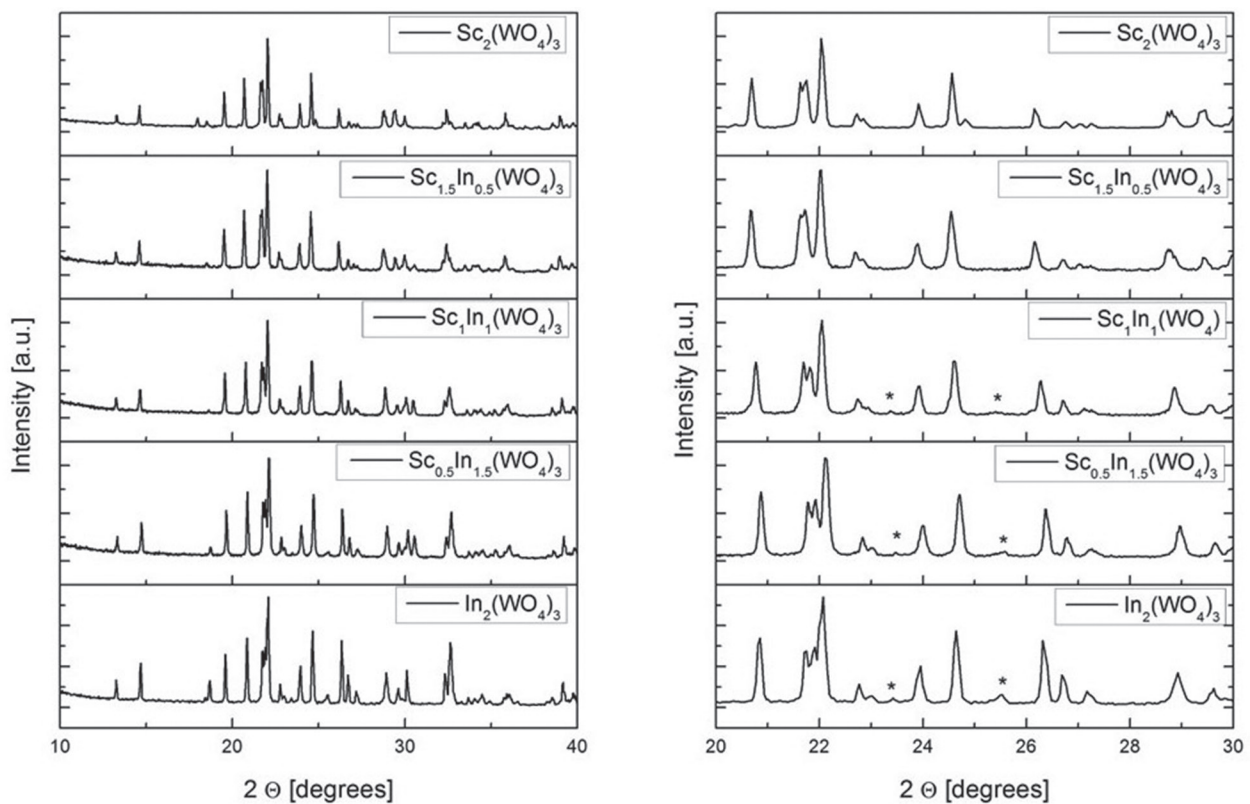


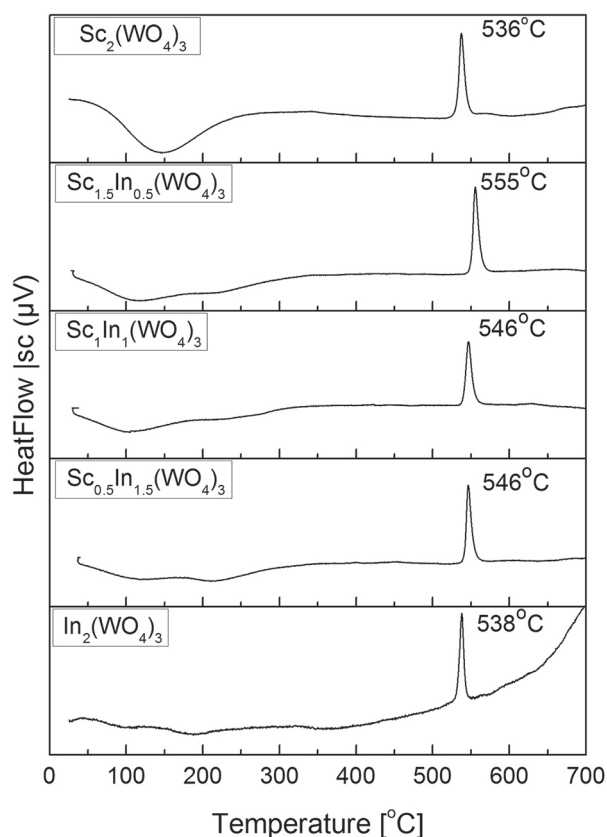
Fig. 1. a) X-ray patterns of non-doped $Sc_{2-x}In_x(WO_4)_3$ ($x = 0\div 2$) precipitates obtained at different pH value and thermally treated at 900 °C for 3 h; b) The same X-ray pattern (narrow scale) with marked picks (*) corresponding to the monoclinic symmetry with space group $P2_1/a$ in the case of $x = 1, 1.5$ and 2 .

Table 1. Cell parameters a, b, c, beta and cell volume, V of non-doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ (1–5) and of doped $\text{Sc}_{2-x-y}\text{In}_x\text{Cr}_y(\text{WO}_4)_3$ (6–9) solid solutions

No	Solid solution composition	a [Å]	b [Å]	c [Å]	beta (°)	V [Å ³]	Symmetry
1	$\text{Sc}_2(\text{WO}_4)_3$	9.677	13.325	9.586	90	1236.1	Pnca
2	$\text{Sc}_{1.5}\text{In}_{0.5}(\text{WO}_4)_3$	9.688	13.328	9.594	90	1238.3	Pnca
3	$\text{Sc}_1\text{In}_1(\text{WO}_4)_3$	16.355	9.637	19.038	125.38	2446.51	P2 ₁ /a
4	$\text{Sc}_{0.5}\text{In}_{1.5}(\text{WO}_4)_3$	16.357	9.634	19.021	125.32	2445.52	P2 ₁ /a
5	$\text{In}_2(\text{WO}_4)_3$	16.375	9.638	19.039	125.31	2452.15	P2 ₁ /a
6	$\text{Sc}_2(\text{WO}_4)_3$; 3%Cr	9.676	13.324	9.584	90	1235.6	Pnca
7	$\text{Sc}_{1.5}\text{In}_{0.5}(\text{WO}_4)_3$; 3%Cr	9.681	13.317	9.585	90	1235.7	Pnca
8	$\text{Sc}_1\text{In}_1(\text{WO}_4)_3$; 3%Cr	16.361	9.632	19.050	125.45	2445.74	P2 ₁ /a
9	$\text{Sc}_{0.5}\text{In}_{1.5}(\text{WO}_4)_3$; 3%Cr	16.341	9.622	19.010	125.36	2437.65	P2 ₁ /a
10	$\text{In}_2(\text{WO}_4)_3$; 3%Cr	16.350	9.627	19.010	125.31	2441.56	P2 ₁ /a

precipitates of the Cr^{3+} doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ solid solutions were subjected to DTA analysis to determine the temperature of phase crystallization. The results are shown on Fig. 2.

On the DTA curves a strong exothermic effects in the temperature range $536\div 555^\circ\text{C}$ were observed. These effects indicate the beginning of the crystal-

**Fig. 2.** Thermal behavior of the 3% Cr^{3+} doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ ($x = 0\div 2$) amorphous precipitates.

lization processes in the precipitates. It can be seen that due to the more complex chemical composition of intermediate solid solutions ($\text{Sc}_{0.5}\text{In}_{1.5}(\text{WO}_4)_3$, $\text{Sc}_1\text{In}_1(\text{WO}_4)_3$ and $\text{Sc}_{1.5}\text{In}_{0.5}(\text{WO}_4)_3$) their initial temperature of crystallization is higher than that of $\text{Sc}_2(\text{WO}_4)_3$ and $\text{In}_2(\text{WO}_4)_3$. The endothermic effects at $100\div 200^\circ\text{C}$ is related to H_2O release. The parallel TG and gas analyses proved that the weight losses are about 5%. The observed exothermic peaks defined the choice of the temperature of thermal treatment for crystallization of solid solutions.

Our previous investigations on obtaining nanosized particles from $\text{Al}_{2-x}\text{Sc}_x(\text{WO}_4)_3$ [24] and $\text{Al}_{2-x}\text{In}_x(\text{WO}_4)_3$ [23] proved that the main factor determining the growth of the particles is the temperature. We have also established that maintaining the same temperature for more than 1 hour has almost no impact on the particles growing. Therefore, the precipitates of non-doped and Cr^{3+} doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ solid solutions were thermally treated at 550, 650, 700, 800 °C and 900 °C in the course of 1 h. As an example, Fig. 3 shows the XRD data of 3% Cr^{3+} doped $\text{Sc}_{2-x}\text{In}_x(\text{WO}_4)_3$ solid solutions obtained at above mentioned temperatures for 1 h. As can be seen, after treatment at 550 °C the products contain negligible amorphous part, while after treatment at 650, 700, 800 and 900 °C the products are well crystallized. The increasing trend of peak tightening with the increasing the temperature of thermal treatment indicates the increase in average crystallites size. The average crystallite dimensions calculated from the X-ray data of the obtained solid solutions are presented in Table 2.

The average crystallite dimensions of the obtained solid solutions, calculated from the X-ray data, are between 19 and 110 nm, except for $\text{Sc}_2(\text{WO}_4)_3$ (Table 2). The influence of the temperature is weak and even at 900 °C, the average crystallites size reach only 110 nm. Higher in-

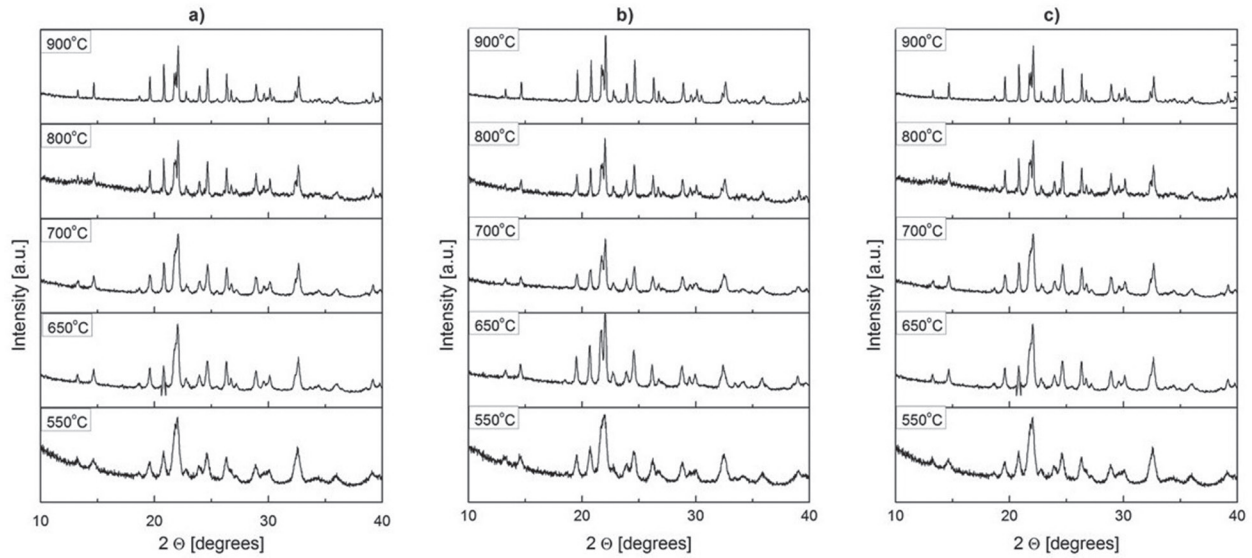


Fig. 3. X-ray powder diffraction patterns of the 3% Cr^{3+} doped precipitates, treated at different temperatures for 1 h: a) $Sc_{0.5}In_{1.5}(WO_4)_3$; b) $Sc_1In_1(WO_4)_3$; c) $Sc_{1.5}In_{0.5}(WO_4)_3$

dium content leads to decrease of crystallites size. The chromium influence depends on the temperature of thermal treatment. The thermal treatment of $Sc_{2-x-y}In_xCr_y(WO_4)_3$ solid solutions at 550, 650, 700 and 800 °C leads to a decrease of the crystallites sizes, while at 900 °C results in increasing the sizes. This result clearly showed that the velocity of phase nucleation in the temperature range 550–900 °C is significantly higher than the speed of nuclei growth and indium and chromium enhance this effect. These results are in good agreement with our previous investigation on tungstate solid solutions from the same class of compounds with a general formula $Me_2(WO_4)_3$ [23–25]. An exception is Cr^{3+} doped $Sc_2(WO_4)_3$ with highest crystallites size –

250 nm at 550 °C, where the velocity of nuclei growth is very high. Due to this, thermal treatment at higher temperature of this sample was not performed. In our earlier investigation, we have established that the combination of high concentrations of Sc^{3+} and Cr^{3+} leads to a strong growth of the crystallite size, (up to the order of 200 nm) [24].

CONCLUSIONS

Nanosized solid solutions from $Sc_{2-x}In_x(WO_4)_3$ and $Sc_{2-x}In_xCr_y(WO_4)_2$ are synthesized for the first time by co-precipitation method. The obtained solid solutions possess orthorhombic symmetry for value

Table 2. Crystallites size dimension [nm] of $Sc_{2-x}In_x(WO_4)_3$ (1–5) and $Sc_{2-x-y}In_xCr_y(WO_4)_3$ (6–10) solid solution at different x and y = 0.06 (3% Cr^{3+}) after thermal treatment at 550, 650, 750, 800 °C and 900 °C

No	Solid solution composition	550 °C	650 °C	700 °C	800 °C	900 °C
1	$Sc_2(WO_4)_3$	65 nm	78 nm	86 nm	90 nm	110 nm
2	$Sc_{1.5}In_{0.5}(WO_4)_3$	30 nm	46 nm	57 nm	77 nm	90 nm
3	$Sc_1In_1(WO_4)_3$	30 nm	40 nm	50 nm	71 nm	89 nm
4	$Sc_{0.5}In_{1.5}(WO_4)_3$	29 nm	38 nm	45 nm	71 nm	88 nm
5	$In_2(WO_4)_3$	27 nm	37 nm	41 nm	55 nm	77 nm
6	$Sc_2(WO_4)_3$: 3 at% Cr^{3+}	250 nm				
7	$Sc_{1.5}In_{0.5}(WO_4)_3$: 3 at% Cr^{3+}	28 nm	45 nm	55 nm	80 nm	110 nm
8	$Sc_1In_1(WO_4)_3$: 3 at% Cr^{3+}	25 nm	38 nm	43 nm	70 nm	110 nm
9	$Sc_{0.5}In_{1.5}(WO_4)_3$: 3 at% Cr^{3+}	24 nm	36 nm	40 nm	68 nm	98 nm
10	$In_2(WO_4)_3$: 3 at% Cr^{3+}	19 nm	30 nm	38 nm	55 nm	85 nm

of $x = 0$ and 0.5 and monoclinic one for value of x greater than 1. Considering the results from the analyses, advantages of co-precipitation method is obvious. It gives the opportunity to obtain nanosized solid solutions in the temperature range 550 ± 800 °C. The crystallites size depends on the ratio of Sc^{3+} , In^{3+} and Cr^{3+} , and on the temperature of thermal treatment and are between 19 and 110 nm. The synthesized solid solutions give opportunity for systematic analyses of properties important for their application, defined by the difference of the two structures.

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СИНТЕЗ НА НАНОРАЗМЕРНИ ЧИСТИ И ДОТИРАНИ С Cr ТВЪРДИ РАЗТВОРИ
С ОБЩА ФОРМУЛА Sc_{2-x}In_x(WO₄)₃

А. С. Йорданова^{1*}, Р. С. Йорданова¹, В. С. Николов¹,
Й. И. Косева¹, П. Т. Цветков¹

¹ *Институт по обща и неорганична химия, Българска академия на науките, ул. „Акад. Г. Бончев“,
бл. 11, 1113 София, България*

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(Резюме)

За първи път бяха синтезирани, наноразмерни твърди разтвори с обща формула Sc_{2-x-y}In_xCr_y(WO₄)₃, за стойности на $x = 0 \div 2$ и $y = 0,02$ и $0,06$, по метода на химичното съутаяване. Бяха проведени фазови и структурни изследвания. Чрез рентгенофазов анализ беше установено, че само чрез стриктно поддържане на рН между 2,7 и 5,7 могат да се получат монофазни продукти. На стайна температура, твърдите разтвори са с орторомбична симетрия за стойности на $x = 0$ и $0,5$ и с моноклинна за стойности над $1,0$. Бяха пресметнати параметрите на елементарната клетка на всички твърди разтвори. Чрез ДТА бяха определени условията на термично третиране на аморфните утайки. Средните размери на кристалитите бяха изчислени от рентгенограмите и са със стойности между 19 и 110 nm. Беше установено, че размерът на кристалитите зависи от температурата на термично третиране и от съотношението между скандий и индий.