

Spark plasma sintering of In_2O_3 -based ceramic targets

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The influence of the main operating factors (applied pressure, temperature and duration of isothermal holding) on the SPS-consolidation processes of dispersed submicron oxide powder compositions with different indium oxide content, as well as the kinetics of compaction and the microstructure of the ceramics formed in this case, was studied. The aim of the work was to obtain relatively high-density ceramics of three compositions relevant for practical application: In_2O_3 - SnO_2 in a weight ratio of 9:1, In_2O_3 - ZnO in a weight ratio of 9:1 and In_2O_3 - Ga_2O_3 - ZnO in a weight ratio of 1:1:1. It was found that during spark plasma sintering of all three compositions there is a certain optimum temperature of isothermal holding (950 °C), above which the density begins to decrease with a significant loss of mass of the initial sample, caused by the intensification of the process of thermal decomposition of indium oxide. Optimization of sintering modes was performed, as a result ceramic targets with a diameter of 50 mm and a relative density of at least 94% of the theoretical density for each composition were obtained. Test magnetron sputtering of the obtained targets showed high discharge stability, without any signs of arcing, which indicates high homogeneity of their composition and properties.

Keywords: SPS, ceramics, transparent conducting oxide, In_2O_3 , ITO, IZO, IGZO, target, magnetron sputtering

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Introduction

Due to high electrical and optical characteristics thin films based on indium oxide are now widely applied as transparent electrodes in various optoelectronic devices designed to operate in a visible range [1–4]. Thus, the transparent electrodes based on Sn-doped In_2O_3 (ITO, Indium Tin Oxide) are still a standard for half a century for traditional devices (displays, Si-based solar panels) [1,3,5,6]. In turn, low-temperature amorphous In_2O_3 films with a Zn impurity (IZO, Indium Zinc Oxide) are more preferable for forming the transparent electrodes in „flexible“ electronics devices on polymer substrates [4,7]. Quite promising materials for the transparent electrodes also include some other compounds based on In_2O_3 doped with various elements [8,9].

But the transparent electrodes are not the only field of application of indium oxide. Substitution of silicon with In-containing double (IZO) and triple (IGZO, Indium-Gallium-Zinc Oxide) oxides that are characterized by high field mobility, in channels of active-matrix TFT structures made it possible to increase display resolution as well as to extend a range of their brightness and contrast [10,11].

Functional In_2O_3 -based films are produced by various types of methods of physical and chemical deposition [1,12,13]. As a whole of technological and commercial factors, the main industrial method of forming functional optoelectronics layers is a method of magnetron sputtering

of ceramic targets of a required composition [14,15]. At the same time, some particular requirements are imposed on the ceramic targets used for industrial deposition of conductive and semiconductor oxide films and it is absolutely necessary to correspond to them for stable production of high-quality heterogenous films. In particular, the ceramic target shall have high purity of the composition, an isomorphic microcrystalline structure and low porosity [16]. These properties provide homogeneity of a process of formation of the thin film due to reduction of probability of arcing during target sputtering and increase duration of its stable operation [17,18]. For the case of the In_2O_3 -based ceramic targets, the high density and homogeneity of the composition is achieved by using ultrafine powders [16], applying costly process equipment and long multi-stage technological processes (isostatic compaction, autoclave annealing in controlled media, etc.) [19].

However, R&D studies in the field of oxide electronics often imply routine optimization of the compositions of the sputtered targets. Therefore, in laboratory conditions it is important to have an express method that provides fast productions of a series of small ceramic targets of an acceptable density (above 90% of a theoretical density) of the various composition with at most exact preservation of an initially downloaded component ratio. This method may be a method of spark plasma sintering (SPS). SPS consolidation is characterized by a temperature and duration

of the high-temperature phase, which is much smaller as compared to the other methods, thereby generally facilitating obtaining of a dense fine-grained ceramic with preservation of the elementary and phase composition of an initial powder mixture [20].

The present study is aimed at demonstrating the prospects of using the SPS method as a fast approach for laboratory production of compact In_2O_3 -based ceramic targets of the required chemical composition and the acceptable density. In the pursuit of this purpose, we have investigated the influence of main acting factors of the SPS method (applied pressure, the temperature and duration of isothermal holding) and the composition of the initial powder compounds on a compaction process and a microstructure of the In-containing ceramic being formed.

1. Materials and research methods

The study has used commercial micropowders In_2O_3 (purity 99.99%, LLC „Rare Metals Plant“, Russia), SnO_2 (purity 99.95%, LLC „Khimsnab“, Russia), ZnO (purity 99.999%, Testbourne LTD, Great Britain) and Ga_2O_3 (purity 99.99%, LLC „Rare Metals Plant“, Russia). Table 1 shows the composition of the prepared initial mixtures.

Homogeneity of the powder mixture was achieved by nine-hour mixing in the argon atmosphere in a ball mill at a 4:1 ratio of a grinding bodies weight ($\text{ZrO}_2\text{:Y}$) to an oxide mixture weight and a drum rotational speed of 60 rpm. Every three hours of activation the powder mixture was averaged by threefold sieving through a sieve with meshes $100 \times 100 \mu\text{m}$ and then was again submersed into a tank for further mixing. Then the powder mixtures were atmospherically annealed in a muffle furnace SNOL12/16 (LLC „Thermoceramics“, Russia) in an alumina container at 750°C for 10 h. After annealing, the powders were additionally sieved through the sieve with meshes $500 \times 500 \mu\text{m}$ and used to produce composite ceramic targets.

Spark plasma sintering of the powders was performed in a laboratory SPS setup of our own design, whose technical characteristics and principal specific feature were previously described [21]. Disk-like ceramic samples were produced by SPS consolidation of powder samples of the respective compositions of the 55 g weight in a graphite mold with a matrix internal diameter of 51 mm. The ceramics were sintered in forevacuum conditions at residual

Table 1. Initial composition of the prepared powder oxide mixtures

Ceramic	Weight of mixture components, g			
	In_2O_3	SnO_2	ZnO	Ga_2O_3
ITO	504	56	–	–
IZO	504	–	56	–
IGZO	187	–	187	187

chamber pressure $P_0 \leq 1 \text{ Pa}$. For all the experiments, a rate of capturing the isothermal holding temperatures was unchanged to be $50^\circ\text{C}/\text{min}$. The sintering modes were optimized by the main acting factors of the SPS method — pressure P_{SPS} applied during sintering, the temperature T_{SPS} and duration t_{SPS} of isothermal holding. Upon completion of each SPS cycle, the matrix with the consolidated sample was cooled down in vacuum at relieved external pressure.

After being extracted from the mold, the disk-like ceramic samples were ground to remove graphite inclusions of mold accessories from their surface using a carbide-silicon powder (grade F120, LLC „Litpromabraziv“, Russia). During this procedure, at least 0.5 mm of a subsurface ceramic material was removed from each of flat sides of the sample. Then the samples were ultrasonically cleaned and dried at 150°C .

An apparent ceramic density ρ_{exp} was determined in full compliance with a procedure determined by GOST 2409–2014 [22] using an electronic analytical balance Adventurer ARC 120 (OHAUS, USA). The true density for the multi-component compositions is calculated according to a mixture rule by the formula

$$\rho_{\text{theor}}^{-1} = \sum X_i / \rho_i,$$

where X_i is a weight portion of the i -th mixture component, and ρ_i is its density. According to reference data, the densities of In_2O_3 , SnO_2 , ZnO and Ga_2O_3 were taken to be 7.18, 7.01, 5.61 and $5.88 \text{ g}/\text{cm}^3$, respectively.

Morphology of ceramic cleavage was studied using a scanning electron microscope LEO 1450 (Carl Zeiss, Germany). The phase composition of the ceramics was studied using a powder diffractometer PANalytical X'PERT PRO MPD (Malvern Panalytical BV, the Netherlands) with a source of $\text{Cu-K}\alpha$ radiation within the range $2\theta = 15 - 75^\circ$.

2. Results and their discussion

2.1. Optimization of the sintering modes

In terms of the influence of initially-nonconductive powder materials on the SPS-consolidation process, the main active process factor is the temperature T_{SPS} . It is followed by pressure P_{SPS} and duration of isothermal holding at the sintering temperature t_{SPS} [20,23]. Therefore, the process of sintering of ITO, IZO and IGZO was optimized by these three factors in the same sequence. The mode was considered to be optimal when the sample density was the highest with preservation of the initial weight of the green body.

Table 2 shows data on the dependence of the apparent ceramic density ρ_{exp} on the isothermal holding temperature with the unchanged values $t_{\text{SPS}} = 300 \text{ s}$ and $P_{\text{SPS}} = 30 \text{ MPa}$ for all the samples. It can be seen that with the smaller content of In_2O_3 in the mixture (the IGZO ceramic) compaction starts at earlier stages — at $T_{\text{SPS}} = 850^\circ\text{C}$ a relative density determined as

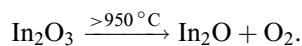
$$\rho\% = (\rho_{\text{exp}} / \rho_{\text{theor}}) \cdot 100\%$$

Table 2. Influence of the isothermal holding temperature T_{SPS} on the apparent density of the ceramic samples ($t_{\text{SPS}} = 300$ s and $P_{\text{SPS}} = 30$ MPa)

Ceramic	$\rho_{\text{theor}}, \text{g/cm}^3$	$\rho_{\text{exp}}, \text{g/cm}^3$			
		$T_{\text{SPS}} = 850^\circ\text{C}$	$T_{\text{SPS}} = 900^\circ\text{C}$	$T_{\text{SPS}} = 950^\circ\text{C}$	$T_{\text{SPS}} = 1000^\circ\text{C}$
ITO	7.16	5.00	6.24	6.88	6.80*
IZO	6.98	4.85	6.03	6.50	—**
IGZO	6.21	5.27	5.69	5.81	—**

Note. * it is recorded that the weight of the green body is significantly lost ($\approx 3\%$); ** — it is recorded that sample integrity is violated after being extracted from the mold.

reached 85% in the IGZO sample, whereas the relative density of the ceramics ITO and IZO did not exceed 70%. An increase of the temperature T_{SPS} to 950°C positively affects the compaction process for all the three compositions — their relative density $\rho_{\%}$ reaches the values much higher than 90%, wherein the most intense increase of the density is observed in the ITO sample, in which the value $\rho_{\text{exp}} = 6.88 \text{ g/cm}^3$ ($\rho_{\%} = 96.1\%$) was reached when $T_{\text{SPS}} = 950^\circ\text{C}$. With the further increase of T_{SPS} to 1000°C , the ITO ceramic exhibits reduction of the density, which is accompanied by loss of weight by $\sim 3\%$, while the ceramics IZO and IGZO collapsed with delamination when being extracted from the mold. Both the facts indicate a significant contribution by gas-formation processes at this temperature [24]. One of the possible sources of gaseous products is a process of high-temperature sublimation of a matrix material of the ceramics by the following reaction:



This thesis can be supported by a previous result of ceramic synthesis in the Zn-Ga-O system at the same SPS setup in similar process modes [25]. This experiment showed that the optimal temperature for this oxide system was higher and was $T_{\text{SPS}} = 1050^\circ\text{C}$. Only when $T_{\text{SPS}} > 1050^\circ\text{C}$ the ZnO:Ga ceramic exhibited reduction of the density due to thermal sublimation of the ceramic components.

Thus, an optimal temperature of spark plasma sintering of the submicron powder mixtures based on indium oxide can be believed to be $T_{\text{SPS}} = 950^\circ\text{C}$, above which a process of indium oxide decomposition intensifies. When being accumulated in pores, the gaseous products at this being formed prevent their collapse by generating a branched microporous structure, which at certain conditions causes its destruction after relieving the external pressure.

Table 3 shows data on the dependence of the apparent ceramic density on compaction pressure P_{SPS} when $t_{\text{SPS}} = 300$ s and at the optimal $T_{\text{SPS}} = 950^\circ\text{C}$. It can be noted here that the increase of pressure to $P_{\text{SPS}} = 40$ MPa results in an increase of the apparent ceramic density for all the three compositions. However, with a further increase of pressure to the value $P_{\text{SPS}} = 50$ MPa all the ceramics

collapsed when being extracted from the mold, wherein the collapse was characterized by delamination, which can also be related to hindrance of exit of the gaseous products of indium oxide decomposition out of a volume of the sintered ceramic.

A result of investigation of the influence of duration of isothermal holding t_{SPS} on the apparent density of the ceramics with the various composition when the values of the temperature (950°C) and pressure (40 MPa) are optimized is generalized in Table 4. According to the Table 4, the increase of duration of holding t_{SPS} from 0 to 300 s results in an increase of the density of all the ceramics. However, when $t_{\text{SPS}} > 300$ s a nature of dependence of the apparent density ρ_{exp} on duration of the SPS process is already different from composition to composition. If the IGZO sample exhibits a continuous increase of ρ_{exp} with an increase of holding time up to $t_{\text{SPS}} = 600$ s, then when $t_{\text{SPS}} > 300$ s the samples ITO and IZO exhibit slight reduction of ρ_{exp} , which is also accompanied by loss of the weight of the consolidated material by $\sim 1\%$. Based on the fact that the samples ITO and IZO initially contain indium oxide almost in three times higher as compared to the IGZO composition, reduction of the apparent density, which is observed in ITO and IZO with the increase of duration of isothermal holding to 600 s, can also be related to partial thermal sublimation of indium oxide, which takes place when $T_{\text{SPS}} = 950^\circ\text{C}$.

Table 3. Influence of pressure P_{SPS} on the apparent density of the ceramic samples ($t_{\text{SPS}} = 300$ s and $T_{\text{SPS}} = 950^\circ\text{C}$)

Ceramic	$\rho_{\text{exp}}, \text{g/cm}^3$		
	$P_{\text{SPS}} = 30$ MPa	$P_{\text{SPS}} = 40$ MPa	$P_{\text{SPS}} = 50$ MPa
ITO	6.88	6.92	—*
IZO	6.50	6.56	—*
IGZO	5.81	5.87	—*

Note. * — violations of integrity of the sample (delamination) after being extracted from the mold.

Table 4. Influence of duration of isothermal holding t_{SPS} on the apparent density of the ceramic samples ($T_{\text{SPS}} = 950^\circ\text{C}$ and $P_{\text{SPS}} = 40\text{ MPa}$)

Ceramic	$\rho_{\text{exp}}, \text{g/cm}^3$				
	$t_{\text{SPS}} = 0\text{ s}$	$t_{\text{SPS}} = 30\text{ s}$	$t_{\text{SPS}} = 100\text{ s}$	$t_{\text{SPS}} = 300\text{ s}$	$t_{\text{SPS}} = 600\text{ s}$
ITO	6.37	6.74	6.81	6.92	6.87*
IZO	5.86	6.22	6.34	6.56	6.52*
IGZO	5.35	5.73	5.80	5.87	5.89

Note. * is one-percent loss of the weight relative to the initial subsample.

Table 5. Optimal modes of SPS synthesis of the In-containing ceramic targets of the acceptable density

Ceramic	Acting factors of SPS			Characteristics of the target	
	$T_{\text{SPS}}, ^\circ\text{C}$	$P_{\text{SPS}}, \text{MPa}$	t_{SPS}, s	$\rho_{\text{exp}}, \text{g/cm}^3$	$\rho\%, \%$
ITO	950	40	300	6.92	96.6
IZO			300	6.56	94.0
IGZO			600	5.89	94.8

2.2. Microstructure and properties of the ceramics

Thus, based on the performed studies, we have determined the optimal modes of SPS synthesis, which provide the In-containing oxide ceramics with the relative density $\rho\%$ of at least 94% of a design value for this composition (Table 5). After that, we have studied the microstructure of these samples.

Fig. 1 shows microphotos of cleavages of the dense ceramic samples. It can be seen that the ITO ceramic (Fig. 1, *a*) has a dense homogeneous structure with a small number of closed pores of sizes from 1 to $10\ \mu\text{m}$, which occupy less than 5% of a studied area. In turn, the cleavages of the ceramics IZO and IGZO (Fig. 1, *b, c*) have a less homogeneous structure and their porosity is of a focal nature. The number of pores and their total area on the microphotos for IZO and IGZO are higher than for ITO, thereby agreeing with hydrostatic weighing data.

We believe that the observed differences in the morphology of the cleavages of the synthesized ceramics can be related both to specific features of agglomeration of particles of the ceramic components when preparing the powder mixtures as well as diversity of interaction (dissolution, phase formation) of the oxides of Sn, Zn and Ga with the matrix indium oxide during SPS consolidation.

Indeed, according to XRD-analysis data, upon completion of 300-second SPS consolidation, the ITO ceramic is a single-phase material — its diffraction pattern exhibits only reflections of a cubic phase of In_2O_3 (Fig. 2, an upper spectrum). It indicates full dissolution of a Sn impurity in the In_2O_3 matrix. At the same time, the diffraction patterns of IZO (Fig. 2, a lower spectrum) and IGZO (Fig. 3)

indicate a two-phase state of the ceramics. After 300-second SPS consolidation, in addition to the In_2O_3 main cubic phase, the IZO ceramic has an impurity phase of double oxide — one of the representative of a homologous series $\text{In}_2\text{Zn}_m\text{O}_{3+m}$, while in IGZO obtained when $t_{\text{SPS}} = 600\text{ s}$ the InGaZnO_4 main phase and the secondary phase of the ZnGa_2O_4 spinel coexist. Biphasity of the ceramics IZO and IGZO can be explained by a limit of zinc solubility in In_2O_3 , which is smaller in relation to Sn and Ga (according to the study [26], it does not exceed several atomic percent and heavily depends on the composition of the synthesis atmosphere).

Based on the optimization results, for each composition we have obtained the composite ceramic targets of the diameter of 50 mm with the relative density of at least 94%. Test *dc* magnetron sputtering of the produced targets showed high discharge stability without arcing. Fig. 4 shows a photo of appearance of the ceramic targets of the various composition, which are produced at the optimized SPS modes, after test sputtering. It can be seen that all the three ceramic targets are characterized by a dark color. This change of the color of the SPS consolidated material confirms both the fact of high-temperature mutual dissolution of the ceramic components as well as the fact that the ceramics were synthesized under strong O-poor condition. A deviation of the ceramic composition towards some oxygen deficiency was also confirmed by the fact that they were characterized by low electrical resistance, which made it possible to further sputter them in the *dc*-mode. Based on results of two-hour test sputterings, all the targets preserved their integrity and ring-like erosion zones formed on their surface during sputtering are visually homogeneous,

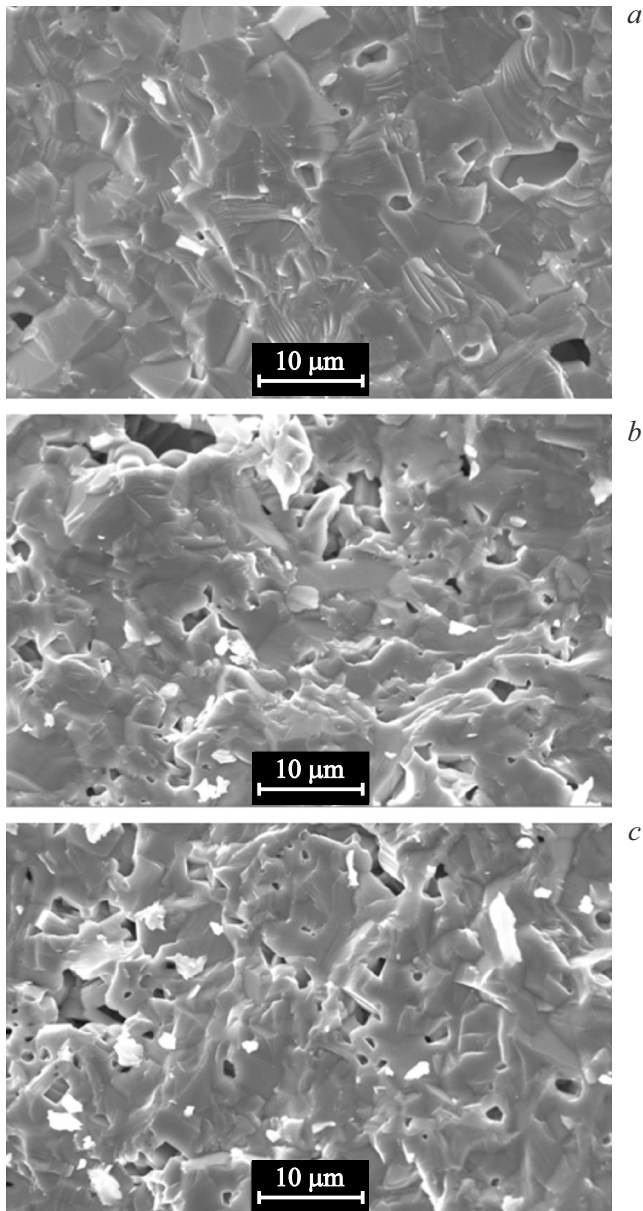


Figure 1. SEM-microphotos of cleavages of the In-containing ceramics synthesized at the optimized values of the SPS mode: *a* — ITO, *b* — IZO, *c* — IGZO.

have not nodules and arcing traces, which indicated high homogeneity and stability of the process of their sputtering.

In conclusion, we would like to note that based on analysis of the results obtained for SPS consolidation of the micron powders and comparison of the results with literature data [20,27–29] we plan some further studies aimed at achieving an ultra-dense state in the In-containing ceramic during its SPS consolidation. In our opinion, first of all, it is necessary to search for methods of „locking in“ hemioxide In_2O that is formed within the ceramic volume at the high temperatures. The studies will include both optimization of the process of preparing the initial powder mixtures [26] as well as improving temperature

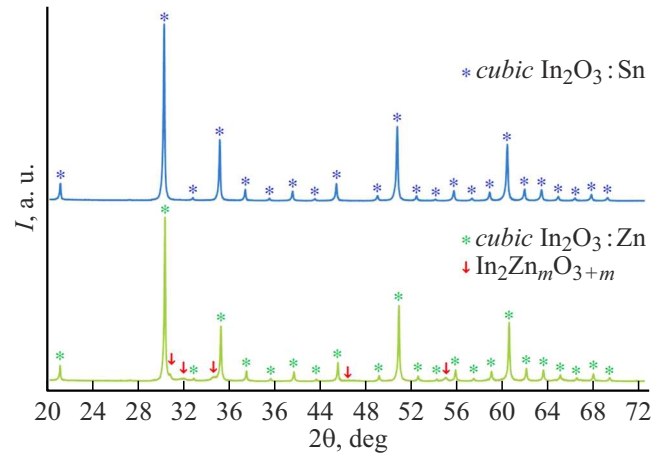


Figure 2. XRD patterns of the ceramics ITO and IZO synthesized at the optimized values of the SPS mode.

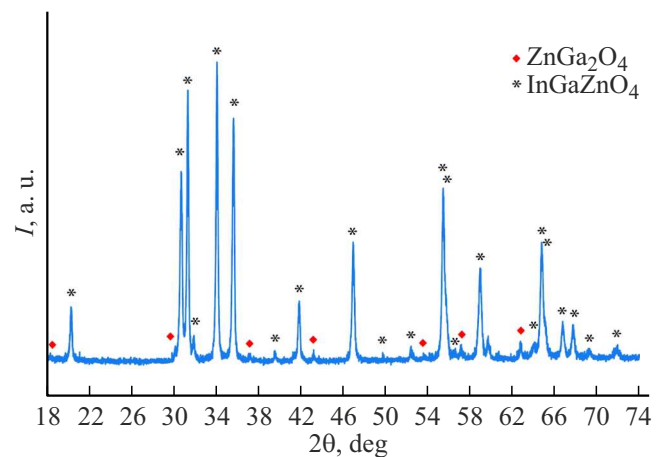


Figure 3. XRD pattern of the ceramics IGZO synthesized at the optimized values of the SPS mode.

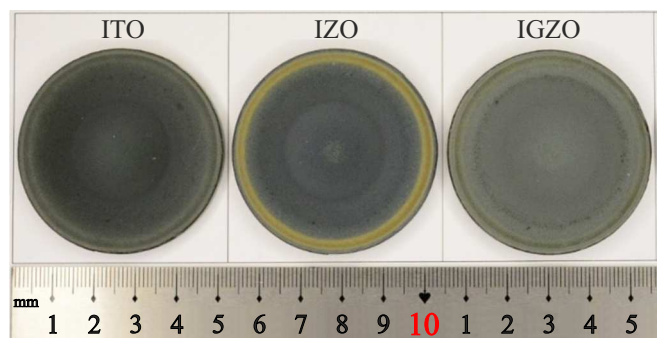


Figure 4. Appearance of the ceramics after grinding the surface and *dc*-magnetron sputtering in the argon atmosphere.

modes of their consolidation [27], including by varying a heating rate and introducing additional isothermal holding plateaus [28].

Conclusion

We have studied the influence of the main acting factors of the method of spark plasma sintering (applied pressure, the temperature and duration of isothermal holding) on processes of consolidation of particles of the submicron oxide powder compounds $\text{In}_2\text{O}_3\text{-SnO}_2$ (in a weight ratio — 9:1), $\text{In}_2\text{O}_3\text{-ZnO}$ (9:1) and $\text{In}_2\text{O}_3\text{-Ga}_2\text{O}_3\text{-ZnO}$ (1:1:1)

The following conclusions may be drawn from the obtained results.

1) intensification of the processes of decomposition and sublimation of indium oxide during spark plasma sintering of the powder mixtures with its high content in vacuum in the graphite molds limits the maximum allowable temperature of the process at the level of 950 °C;

2) loss of the weight in the consolidated ceramics, which is related to sublimation of In_2O_3 , imposes limitations on duration of isothermal holding as well. For the compositions containing up to 90 wt.% In_2O_3 , optimal duration of isothermal holding at 950 °C is limited by 300 s.

Based on results of the performed optimization, for each of the compositions of the initial micro-powder mixtures we have obtained ceramic samples with a relative density $\rho\%$ of at least 94%. Judging by their demonstrated characteristics, these samples can be successfully used as sputtered targets for *dc*-magnetron deposition of transparent conductive and semiconductor layers ITO, IZO and IGZO.

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Conflict of interest

The authors declare that they have no conflict of interest.

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