Ceramics on the basis of zinc orthostannate

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Abstract. This article presents the results of a study of the process of synthesis of zinc orthostannate powder by the traditional chemical method of mixing the initial zinc and tin oxides in a stoichiometric ratio of 2:1. The phase composition of this mixture after calcination at temperatures of 850-1250°C was studied by x-ray diffraction. The microstructure of the powder was studied by scanning electron microscopy. The optimal temperature for the synthesis of zinc orthostannate is 1250°C. The synthesized porous ceramics on the basis of zinc orthostannate (\(P_\rho = 38.6\) %, \(\rho = 3.84\) g/cm\(^3\))

1. Introduction
A promising material in the field of solar energy, gas sensitivity sensors is zinc orthostannate (\(\text{Zn}_2\text{SnO}_4\)), which has excellent properties, namely, a large band gap (\(E_g = 3.35\) eV), thermodynamic stability, high electron mobility, electrical conductivity and low absorption in the visible range. The components are quite cheap and affordable, which makes the introduction of zinc orthostannate in the field of solar energy as a solid-state transparent electrode particularly promising [1, 2].

2. Experimental
Zinc (II) and tin (IV) oxides were taken as the starting materials for the synthesis, which were mixed in a jar roller in a stoichiometric ratio of 2:1 with corundum grinding bodies in an acetone medium for 3 hours at 60 rpm. After mixing, the mass was dried in a fume hood at 85°C for a day. The dried powder mass was twice ground through a sieve with a cell size of 0.5 mm. Then the oxide mixtures were calcined in the temperature range from 850 to 1250°C in increments of 50°C, after which x-ray diffraction of the powder at each of the synthesis temperatures was performed.

Next, the calcined powder with 5% PVA was formed in a prismatic mold with a size of 40×6×6 mm at a pressure of 100 MPa and dried in air 20-25°C for a day. Firing was carried out in an atmospheric silicate furnace at a temperature of 1250°C with exposures of 1, 2 and 3 hours.

3. Results and discussion
Based on the conducted set of experiments, we can draw a number of conclusions about the obtained materials.

The method of preparing the source materials in the ZnO-SnO2 system is of great importance. According to the results of x-ray diffraction (figure 1), when calcining from 850 to 1250°C with an increase in temperature, the main reflection reflex of SnO2 decreases, and at a temperature of 1200°C, it disappears, which indicates that it is tin oxide completely reacted. And at 1250°C, the complete formation of the crystal lattice of zinc orthostannate is observed. Thus, the synthesis of the solid solution was performed at this temperature.
Roasting performed from a powder of zinc orthostannate of the samples was conducted at a temperature of 1250°C with an exposure time of 1, 2 and 3 hours. The best properties are found in the material burned at two-hour exposure.

According to the results of microscopy (figures 2-5) of synthesized powders obtained at temperatures of 950-1250°C with a step of 50°C, the formation of a two-phase powder is visible. The first phase is fine-grained, with an average particle size of less than 1 microns; the second phase is well-crystallized, coarse-grained, with an average particle size of 8-10 microns. It is also seen that with increasing temperature, the amount of fine fraction of tin oxide decreases. Tin oxide particles form aggregates that subsequently form crystals. When the synthesis temperature approaches 1200°C, the complete disappearance of SnO$_2$ particles occurs, this is just confirmed by the previously conducted x-ray diffraction.
Figure 2. Microstructure of the powder calcined at 950°C (a) and 1000°C (b).

Figure 3. Microstructure of the powder calcined at 1050°C (a) and 1100°C (b).
Figure 4. Microstructure of the powder calcined at 1150°C (a) and 1200°C (b).

Figure 5. Microstructure of the powder calcined at 1250°C.

Figure 6. Effect of holding time during firing on the average density of samples and open porosity of samples.
Based on the results of determining the average density and open porosity, it follows that it is impossible to obtain a transparent electrode of zinc orthostannate in a standard way, without the use of modifying additives, for example.

4. Conclusion

Based on the work carried out, it was found that when obtaining a powder of zinc orthostannate by mixing the initial oxides and further calcining this mixture at temperatures of 850-1250°C, x-ray diffraction showed that the optimal temperature is 1250°C. It was also found that the material with the best characteristics was obtained by firing the formed mass at a temperature of 1250°C and exposure time of 2 hours. The obtained porous ceramics on the basis of zinc orthostannate (Po = 38.6 %, \( \rho = 3.84 \text{ g/cm}^3 \)).

To obtain the best sintering powders of zinc orthostannate, it is necessary to use a chemical synthesis method at lower temperatures, as well as the introduction of additives for solid-phase sintering of d-element oxides, which will allow to intensify the sintering process.

References

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