SOL-GEL SYNTHESIS AND INVESTIGATION OF UN-DOPED AND EU-DOPED STRONTIUM

ALUMINATE SR3AL2O6

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СИНТЕЗ И ИССЛЕДОВАНИЕ АЛЮМИНАТА СТРОНЦИЯ SR₃AL₂O₆, АКТИВИРОВАННОГО ИОНАМИ ЕВРОПИЯ

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Аннотация. Алюминат стронция был получен методом золь-гель. Использовали термическую обработку в сушильном шкафу для сушки геля. Для получения кристаллического продукта кубической модификации все образцы прокаливали в муфельной печи при температурах до 1000 ° С. Морфологию поверхности прекурсора исследовали при растрового электронного микроскопа Hitachi TM3000. На основании данных рентгенофазового анализа сделали вывод об образовании алюмината стронция состава $Sr_3Al_2O_6$ с незначительными примесями фазы $SrAl_4O_7$. Золь-гель методом был получен люминофор состава $Sr_{2,8}Eu_{0,2}Al_2O_6$. Экспериментально было подтверждено свечение красного цвета полученного люминофора.

Introduction. In recent years, phosphors (i.e., solids and substances that are capable of luminescence under the action of excitation) have been widely applied in different areas of production: emergency lighting systems, special ceramics, and luminous paints due to their ability to emit a certain spectrum of waves [1]. Bright examples of luminescent compounds are aluminates of alkaline earth metals, particularly strontium aluminate. This chemical compound is characterized by high refractoriness and mechanical strength. Due to the type of crystal lattice and a certain crystallographic environment, strontium aluminate, which is activated by rare-earth ions, is capable of emitting light of various colors (depending on the RDF used) [1]. Currently, several methods for obtaining different phases of the given compound are well-known, however, the formation of aluminate of the $Sr_3Al_2O_6$: Eu structure by sol-gel method has not been sufficiently studied, which has affected the formulation of the task and the aim of the present work. The purpose of this research project includes 1) obtaining strontium aluminate by sol-gel method using citric acid as a polymerizing agent; 2) activation of the obtained strontium aluminate with europium (III) ions; 3) the formation of the phosphor $Sr_3Al_2O_6$: Eu.

Experimental: synthesis of strontium aluminate. We based our data on the phase diagram of SrO-Al₂O₃ system while selecting ratio between starting reagents. On the diagram, $Sr_3Al_2O_6$ is forming at a 3:1 molar ratio of 3SrO: Al₂O₃ oxides. Strontium nitrate $Sr(NO_3)_2$ and aluminum nitrate nonahydrate $Al(NO_3)_3 * 9H_2O$ were used as

147

148 ХV МЕЖДУНАРОДНАЯ КОНФЕРЕНЦИЯ СТУДЕНТОВ, АСПИРАНТОВ И МОЛОДЫХ УЧЕНЫХ «ПЕРСПЕКТИВЫ РАЗВИТИЯ ФУНДАМЕНТАЛЬНЫХ НАУК»

oxidizer and cations source. Citric acid $H_8C_6O_7 * H_2O$ was used as a curing agent. We calculated the required amount of reagents based on a molar ratio of 3: 2: 5 and reduced the mass obtained by a factor of 100:

Using the technical scales, tracing paper, and spatula, we weighed the required quantity of reagents. For solution preparation, we dissolved suspended reagents in water in the following proportions: 6.36 g of Sr (NO3) 2 in 8.996 ml of distilled water; 7.5 g of Al (NO₃)₃ * 9H₂O in 10 ml of water; 10.5 g of H₈C₆O₇ * H₂O in 7.89 ml of water. The resulting solutions were mixed with a glass rod to completely dissolve the solids.

$$SrCO_3 + Al_2O_3 = SrAl_2O_4 + CO_2$$

 $2SrCO_3 + SrAl_2O_4 = Sr_3Al_2O_6 + 2CO_2$

Further, solutions were poured into a single beaker, the magnetic agitator platform was installed, and a small magnet was placed at the bottom of the glass for 1.5 hours. The final sol was transferred to a porcelain dish and heat treated at 130 $^{\circ}$ C in an oven to obtain crystalline strontium aluminate hexagonal modification. Then, the obtained dry residue of rufous color was cooled and ground in a mortar. The final powder was poured into a crucible and placed in a muffle furnace for 3 hours at a temperature of 1000 $^{\circ}$ C. At the end of the period, the final synthesis product was removed from the muffle furnace, cooled and poured into a container for SEM and MRSA [2].

Synthesis of strontium aluminate activated by europium ions. Ratio Sr: Eu: Al: O as 2.8: 0.2: 2: 6 complying with the formula $Sr_{2,8}Eu_{0,2}Al_2O_6$ was chosen for preparation of phosphor-luminescent strontium aluminate activated by europium ions. Oxide Eu_2O_3 was used as a europium containing substance. We calculated the required amount of reagents based on a molar ratio of 2,8: 0,2: 2: 6 and reduced the mass obtained by a factor of 100:

Using the technical scales, tracing paper and spatula, we weighed the required quantity of reagents. We dissolved suspended reagents in water in the following proportions: 5.936 g of Sr (NO₃)₂ in 8.4 ml of distilled water; 7.5 g of Al(NO₃)₃ * 9H₂O in 10 ml of water; 10.5 g of H₈C₆O₇ * H₂O in 7.89 ml of water. Europium oxide (III) 0.176 g was dissolved in nitric acid by stirring and simultaneously heating the mixture on a magnetic stirring plate. Strontium aluminate Sr₃Al₂O₆, activated by Eu-ions, was obtained by the method described above. The final synthesis product was poured into a container for SEM and MRSA [2].

Results and discussion.

Identification of the obtained product of strontium aluminate by SEM, XRF and MRSA method. The phase composition was investigated by X-ray phase analysis. According to the obtained data, we concluded on the formation of strontium aluminate $Sr_3Al_2O_6$ with insignificant impurities of $SrAl_4O_7$ phases (*Fig.* 1).

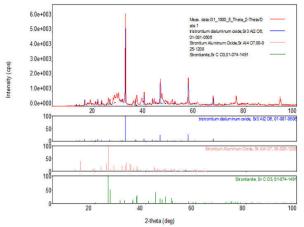


Fig. 1. Spectrum of phases obtained strontium aluminates

ХV МЕЖДУНАРОДНАЯ КОНФЕРЕНЦИЯ СТУДЕНТОВ, АСПИРАНТОВ И МОЛОДЫХ УЧЕНЫХ «ПЕРСПЕКТИВЫ РАЗВИТИЯ ФУНДАМЕНТАЛЬНЫХ НАУК»

According to the SEM, the sample consists of two kinds of particles with different sizes. Large particles are agglomerates of sintering (*Fig.* 2).

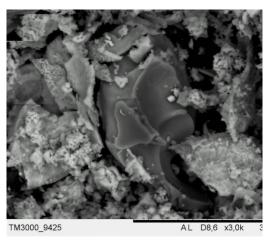


Fig. 2. Morphology of the precursor surface

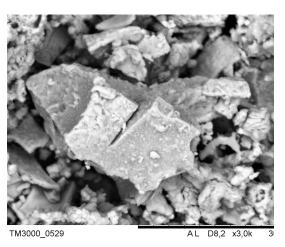


Fig. 3. Morphology of the phosphor surface

According to MRSA, spectral lines of strontium, aluminum, and oxygen were revealed, which indicates the entry of elements into the structure of the precursor.

Identification of the product $Sr_3Al_2O_6$: Eu by SEM and MRSA methods. The morphology of the phosphor surface was investigated by scanning electron microscopy on a SEM Hitachi TM3000. According to the obtained data during the RFA, we concluded on the formation of strontium aluminate activated by europium ions with the $Sr_3Al_2O_6$: Eu structure possessing insignificant impurities. The sample consists of two kinds of particles with different sizes. Large particles are agglomerates of sintering (*Fig.* 3).

According to MRSA, spectral lines of strontium, aluminum, oxygen and europium were detected, which indicates the entry of elements into the phosphor structure.

According to micro-X-ray spectral analysis, a uniform distribution of the activator and elements over the surface was observed.

Conclusion. A single-phase cubic strontium aluminate $Sr_3Al_2O_6$ was obtained by sol-gel method using a thermal treatment in the gel drying step. X-ray phase analysis revealed strontium aluminate $Sr_3Al_2O_6$ with $SrAl_4O_7$ phase impurities. Strontium aluminate $Sr_{2.8}Eu_{0.2}Al_2O_6$ was obtained. According to MRSA, the distribution of elements and the activator on the phosphor surface revealed the occurrence of europium in the structure of strontium aluminate. Red luminescence of the resulting phosphor was observed during its excitation.

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149