Synthesis of materials with a hierarchical structure based on tin dioxide

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SnO₂-based hierarchical structures have been intensively studied because they have large surface area, high surface permeability, low density, low cost, environmental friendliness, and stable physicochemical characteristics [1]. This paper presents a way to create SnO₂-based hierarchical micro-nanostructures with controlled size. The proposed method is based on the use of the SnCl4/EtOH/NH4OH lyophilic film-forming system. Due to the greater stability of the properties of the films obtained from this film-forming system, with prolonged temperature exposure, the service life of the gas sensor is increased. Figure 1 shows the structure of films obtained from the SnCl₄/EtOH/NH₄OH film-forming system with different ratios of ammonium ions to tin ions by the sol-gel method. The photographs were taken with an MPE-11 optical microscope. Figure 1a shows that in the absence of ammonium hydroxide no regular structures are found on the film. When ammonium ions are added, certain structures begin to form, the shape and size of which depend on the relative amount of tin ions N(Sn) and ammonium ions $N(NH_4)$. At $N(Sn) > N(NH_4)$, structures resembling a sixpetalled flower are formed (Figures 1b, 1c, 1d). The average size of the synthesized structures increases from 10 to 40 µm with an increase in the content of ammonium ions. At $N(Sn) \leq N(NH_4)$, the formation of cruciform structures predominates (Figures 1e, 1f). Their size is much larger than flower-like structures and reaches more than 300 µm. When the ratio of ammonium ions to tin ions is 2:1, structures with the largest size are formed.



Figure 1 – The structure of the film obtained from the SnCl₄/EtOH/NH₄OH film-forming system by the sol-gel method, with different amounts of NH₄OH per 100 ml of solution: a) without adding NH₄OH; b) - 0.1 ml; c) - 0.2 ml; d) - 0.4 ml; e) - 0.8 ml; f) - 1.6 ml.

To study the crystal structure of the obtained films, measurements were made on a DRON-6 X-ray diffractometer. The measurement results are shown in Figure 2, in which we have marked the signal from the crystallographic plane $NH_4Cl(110)$ and from the three crystallographic planes $SnO_2(110)$, $SnO_2(101)$ and $SnO_2(211)$.



Figure 2 – X-ray diffraction pattern of the crystal structure of the film obtained from the SnCl₄/EtOH/NH₄OH film-forming system (with 0.8 ml of NH₄OH per 100 ml of solution) by the sol-gel method, measured on a DRON-6 X-ray diffractometer.

The average sizes of crystallites were calculated using the Scherrer formula, which relates the sizes of crystallites to the width of diffraction peaks [2]. The size of SnO_2 crystallites is 3.6 nm, and the size of NH_4Cl crystallites is 109 nm. Thus, SnO_2 and NH_4Cl are involved in the formation of the synthesized hierarchical structures.

This research was funded by the Science Committee of the Ministry of Education and Science of the Republic of Kazakhstan grant number AP19574404.

References

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