ZnO_2/SiO_2 Core/Shell type system prepared via Pechini Method combined with Sol-Gel route monitored by High Resolution TEM.

Alessandro B. S. Garcia (IC)[1], Glenda G. Souza[1] (PG), Ana M. Pires[1*] (PQ).

***anapires@fct.unesp.br**

[1]*Depto. Quím. e Bioq. – FCT – UNESP, R. Roberto Simonsen, 305, Presidente Prudente, São Paulo, Brasil.*

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Abstract

XRD and high resolution TEM images showed that Stöber and Pechini methods combination successfully produced ZnO whurtzita type phase in the presence of spherical amorphous silica.

Introdução

Core/shell type systems involve two or more compounds by using encapsulation process in order to obtain a final material with combined properties. ZnO is frequently used as core because it is a low cost semiconductor applied in light emitting devices in the UV-Vis, with a bandgap around 3.37 eV. In our research group, Pechini method has been used to produce $ZnO@Eu_2O_3$ core/shell luminescent systems of spheroidal nanoparticles, however quite agglomerated [1,2]. In order to generate more dispersed systems, with homogeneous coating, it was decided to include a new layer of SiO_2 before the insertion of Eu_2O_3 shell. Therefore, the objective of the present work is to use high resolution TEM images to monitor the synthesis of ZnO/SiO_2 systems combining the Pechini method and Stöber sol-gel methodology [3] and evaluate its potentiality as a core for subsequent coating with Eu_2O_3.

Results and Discussion

The system containing ZnO/SiO_2 was obtained via Pechini modified method[1,2] combined with sol-gel route based on the Stöber methodology[3]. X-ray diffraction data (XRD), Fig. 1, show the presence of ZnO whurtzita phase (JCPDS-36-1451 file) without displacements as well as a halo in 21.74º characteristic of amorphous silica. From TEM images, Fig. 2, it is possible to observe the formation of spherical silica particles with a diameter around 180 nm. The scattering of the electron beam during TEM image acquisition caused by amorphous silica difficult to ZnO identification in the system; however it is possible to observe individual particles of ZnO out of the spheres whose high-resolution image allowed us to estimate the average interplanar distance value of 2.895 Å, related to the ZnO (100) d_{hkl} plane, Fig. 2(b) inset. It was also possible to observe particles with different contrasts inside of them adhered to spheres surface, with an example in the inset of Fig. 2(b), that can be an indicative of ZnO coated with silica.

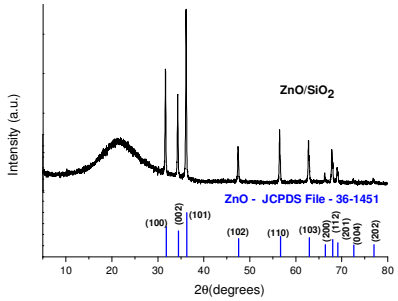

Fig.1 XRD of ZnO/SiO_2 sample.

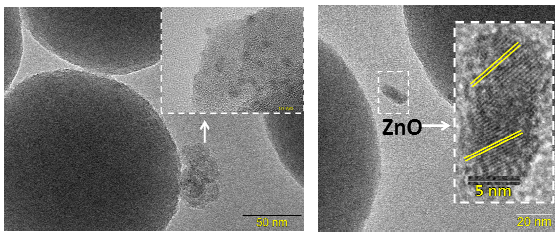

Fig.2. High resolution TEM images of ZnO/SiO_2 sample

Conclusions

XRD data confirms that Stöber and Pechini methods combination produced ZnO whurtzita type phase in the presence of amorphous silica. TEM images, for instance, showed that the majority of the particles are spherical silica ones, with few ZnO nanoparticles dispersed. However, it was also observed regions that give an indicative of ZnO phase coated with silica adhered on the surface of the spheres. In addition there is a strong probability that most of the ZnO particles which could not be viewed are inside of the spheres, since XRD ZnO profile is very well defined and ensure ZnO presence. Thus, the method can be considered appropriate to produce particles more dispersed; however, for subsequent coating with Eu_2O_3 shell, it'll be necessary to reduce the proportion of SiO_2 to evidence ZnO phase.

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