Preparation of ytterbium and erbium 2-methoxyethoxides applicable at the sol-gel synthesis of RE garnets

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Among rare earth (RE) ions, the doping of either Er3+ or Yb3+ is often used in optical materials. When optical materials are prepared by sol-gel process, the dopants are added in different forms depending on the chosen method. However, thanks to the low concentration (tenths of %), the form of starting compound has negligible effect on the quality of a final film. Different situation occurs when rare earth ions are a major part of a host material, such as RE aluminium garnets - e.g. $Yb_3Al_5O_{12}$ has been established as a suitable luminescent oxide. In this case, an inappropriately chosen starting RE compound can cause cracking of the thin films during the thermal treatment of deposited gel layers. An alkoxide sol-gel process is a powerful way at functional oxides preparation and 2-methoxyethoxide metal derivatives are successfully used at films deposition due to their lower sensitivity to hydrolysis.

There are only a few works about the preparation of RE 2-methoxyethoxides; moreover, none of them describes further use of the alkoxide in a sol-gel process. Our aim was not to isolate individual 2-methoxyethoxides but to prepare intermediate solution without any necessary purification, directly applicable at the preparation of Yb/Er/Al alkoxide solution.

Different synthesis methods of preparation were used. The formation of alkoxides was studied by IR spectroscopy. Especially, the changes in the C-O terminal and bridging stretching modes (that are typical for metal alkoxides) were monitored.

Firstly, ytterbium or erbium powders were heated at 126 °C under reflux in 2-methoxyethanol in the presence of $HgCl_2$. Even though the heating time overcame 10 days, the quantitative dissolution was not achieved. When electrolysis was applied, the metals dissolved in 2-methoxyethanol readily. However, as the electrolysis was proceeding, the surface of metal pieces was corroded so that the powder of unreacted metal shed to the solution. Therefore, after electrolysis, it was necessary to filter the suspension. Moreover, the method produces “waste” metal powder and becomes uneconomical. The exchange reaction between i-propoxides and 2-methoxyethanol was fully successful; arising i-propanol was easy to distil out. The ease of this reaction is caused by the sum of several factors - e.g. the lower boiling point of i-propanol or slightly lower pKa of 2-methoxyethanol. The exchange reaction between acetates and 2-methoxyethanol was also studied. In this case, we can confirm an exchanging reaction also by the detection of released acetic acid. Unfortunately, the acid under reflux in the used alcohol forms ester which cannot be distilled out (b.p. 145 °C). The present ester could cause the cracking of a prepared film.

Solutions from exchange reactions are suitable for thin film preparation without any purification. We are going to study the influence of the ester presence on a film microstructure in the further work.