

A NEW TECHNIQUE FOR THE PREPARATION OF $KReO_4$ COMPOUND: THE ACRYLAMIDE SOL-GEL METHOD VIA MICROWAVE AND THE COMPARISON WITH THE SOLID STATE REACTION.

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$KReO_4$ compound was prepared by two different methods: the acrylamide sol-gel method and the solid-state reaction. Powder X-Ray Diffraction (XRD) analysis was used to characterize the process of crystallization. Particle size and morphology of polycrystals were examined by Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM).

In case of solid-state reaction a stoichiometric mixture of metallic Re and K_2CO_3 were ground in an agate mortar in air. XRD patterns of powder resulting mixture showed the Re and additionally the presence of $KReO_4$. Then this powder was treated at 500 °C for 24 h showing a single phase $KReO_4$, which can be related with Powder Diffraction File (PDF) 70-0887. This single phase have a tetragonal unit cell. The resulting powder was pressed into a pellet (13 mm) and annealed in a flux of oxygen at 500 °C during 22 h. XRD pattern shows the presence of $KReO_4$, also the ReO_3 (PDF 40-1155) and a peak corresponding to $d = 3.8526$ that have not be identified. This material was annealed in an air atmosphere at 500 °C during 240 h. XRD patterns in this case showed the $KReO_4$ formation; two values of d were not recognized.

In sol-gel method via acrylamide, the xerogel was obtained by dehydration of the acrylamide during 16 h anneal at 500 °C. XRD patterns showed the presence of the ReO_3 (PDF 40-1155), ReO_2 (PDF 09-0274), $KReO_4$ and $d = 3.8006$. This sample was pressed into a pellet and annealed in an air atmosphere at 505 °C during 70 h. Better ordering was observed in XRD patterns, showing $KReO_4$ compound and three d values unknown.

SEM and TEM observation shows two different scales, micro and nanometric scale, in the crystallization of particles obtained depending on synthesis process.

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