

Structural study of the crystalline phase in Pb-Ca-Ba-Cu-O precursor films grown by spray pyrolysis technique

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Abstract – We present a statistical study of the crystalline phase distribution in Pb-Ca-Ba-Cu-O precursor films grown by spray pyrolysis technique, with thickness and composition suitable to incorporate Hg by the sealed quartz tube technique in a subsequent treatment. The understanding of the dynamics of precursor phases that occur during the preparation of precursor films allow to determine optimal methods of preparation by controlling the deposit and annealing parameters. Chemical composition was obtained from atomic absorption measurements. Crystalline phase identification was performed by XRD technique and the quantification was determined by Rietveld method. Ternary phases were obtained between 835 and 860 °C.

In this work, we present a statistical study of the crystalline phase distribution in precursor films of the type Pb-Ca-Ba-Cu-O, grown by spray pyrolysis system, with thickness and composition suitable to incorporate Hg by the sealed quartz tube technique in subsequent treatment. A series of 9 precursor thin films type Ba-Ca-Cu-Pb-O were deposited on MgO substrates with the spray pyrolysis technique from solutions with different concentrations at different temperatures. Subsequently, the films were under different thermal treatments. Samples were prepared according to an experimental design (Table 1), in order to observe the effect on the variation of these parameters in the composition and the thickness of the thin films obtained. Chemical composition was obtained from atomic absorption measurements. Crystalline phase identification was performed by XRD technique in a Siemens D-5000 diffractometer. Quantification was determined by Rietveld method. A study by Atomic Absorption of the deposit films revealed the absence of nitrates which avoid the formation of impurity phases.

Interdependence between deposition temperature and solution concentration in a first step; and annealing temperature and annealed time in a subsequent thermal treatment, in the relative percentages of each precursor phases was studied applying a fractional factorial design 3^{IV-II} . After annealing we can find mainly ternary phases suitable for the incorporation of Hg. The ternary phases BaPbO₃, Ba₄Pb₃O₁₀ and BaCuO₂ are obtained at 810°C, and between 835°C and 860°C, respectively. The deposition temperature and molar solution have more influence on the thickness of the film, instead of the parameters of the thermal treatment.

Table 1: Design N2: Fractional factorial design 3^{4-2} for the determination of the influence of T_D , T_R , t_R , and molarity on the thickness of the precursor film.

Sample	T_D [°C]	M	T_R [°C]	t_R [hrs]
ra1 / TT1	150	0.005	810	3
ra2 / TT5	250	0.005	835	21
ra3 / TT9	350	0.005	860	12
rb1 / TT6	250	0.010	860	3
rb2 / TT7	350	0.010	810	21
rb3 / TT2	150	0.010	835	12
rc1 / TT4	250	0.015	810	12
rc2 / TT8	350	0.015	835	3
rc3 / TT3	150	0.015	860	21

References

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