Synthesis and microstructural characterization of zirconia scandia system

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In the last years the production of clean energy has been very important replacing or improving the materials to reduce the amount of material, weight, operating temperature, cost, etc. Solid electrolytes are used in the new technologies as Solid Oxide Fuel Cells (SOFCs) where is important to reduce the operation temperature. These cells operate at temperatures between 700 and 850 °C and the priority of lowering the operating temperature of SOFCs have attracted great interest worldwide. One of the greatest barriers to reduce the temperature of operation is the ohmic loss of the electrolyte. Maximize the ionic conductivity of the electrolyte is of significant importance especially in the absence of a variety of materials for electrolytes. Zirconia stabilized with different oxides as used as electrolyte solid in the SOFCs, since some years the most commonly used electrolyte is yttrium stabilized zirconia and recently was introduced the use of stabilized zirconia scandia. It is well known that zirconia has three polymorphic phases: high-temperature cubic phase, tetragonal (intermediate temperature) and low-temperature monoclinic phase. And that the stabilization of phases may be carried out by adding a doping agent, which may be calcium oxide, magnesium, yttrium, cerium, scandium and rare earths; the phase depends on proportions of these oxides and the thermal procedure. The phase diagrams of the calcium,



magnesium and yttrium oxides is known that are well defined, but in the case of zirconia stabilized with scandia, the system only have a small region that include the cubic and tetragonal phases, it does difficult getting these phases. A lot of work has been done through different methods: Gel combustion synthesis [1], co precipitation [2] and ceramic processing in which normally is obtained a rombohedral phase rather than a cubic or tetragonal phases.

In this work we used ceramic processing to obtain zirconia stabilized with scandia. The powders were grinded, calcined, uniaxially pressed and sintered at 1600 and 1650 °C during 3 hours, for tetragonal and cubic phase respectively. Several experiments were performed varying the percentage of scandium oxide (10 to 24 %W) and sintering times to achieve the stabilization of the tetragonal phase. Under the best conditions a target was made and thin films were grown by sputtering. The same procedure was done to the cubic phase, increasing the content of scandium to obtain the cubic phase as shown the difractogram in Figure No. 1a. For this study, TEM cross section samples were prepared by scrapping with a diamond point. TEM images and microanalysis were performed on a JEOL JEM 2200FS with Oxford and HAADF detector. X ray diffraction was done with a Panalytical Xpert PRO MPD diffractometer using a grassing incident X ray beam.

The Figure 2 a) shows a cross section of the tetragonal phase Scandia film stabilized with zirconia and Figure 2b) shows a high resolution image arrangement of atoms for the zirconia stabilized scandia in the tetragonal phase. Figure 1b corresponds to an EDS analysis of a thin film in tetragonal phase.



Through the optimization of the amount of scandium oxide added in the ceramic process it has been possible to obtain a powder tetragonal phase and to maintain it in as a thin film using the sputtering system V3 Intercovamex besides tablets of zirconia in cubic phase. The target with the cubic phase will be elaborated in the future and be used for the deposition of thin films and will be evaluated the ionic conductivity of thin films cubic compared with the tetragonal phase

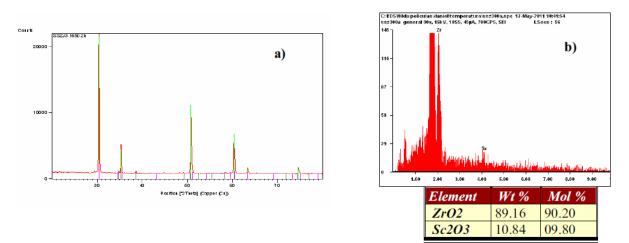


FIG. 1. a) X ray diffraction zirconia cubic and b) EDS thin film onto Si (100) zirconia Scandia system.

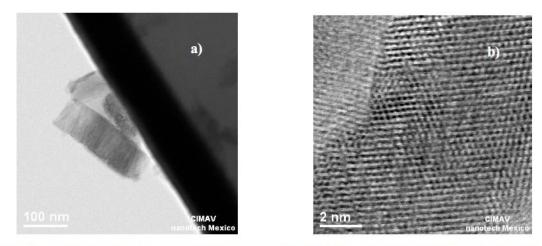


FIG. 2. a) Cross section thin film thickness about 100nm and b) High resolution image of thin film tetragonal phase.



References

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