

Effect of the synthesis conditions of $\text{Ce}_{0.9}\text{Gd}_{0.1}\text{O}_{1.95}$ (10GDC) powder on its morphology and properties of oxygen-conductive ceramics obtained by spark plasma sintering (SPS).

In this work, two 10GDC powders were obtained by thermal decomposition of the same plate-like oxalate precursor under different reaction conditions. The powders with different morphologies were sintered using SPS. Electron microscopy was applied to investigate the ceramic samples obtained; their ionic conductivities were measured and compared.

Nowadays, Gd-doped ceria (abbr. GDC) is a material widely used as a solid electrolyte in solid oxide fuel cells (SOFC). High oxygen conductivity of GDC at intermediate temperatures 500-700°C is favorable for using it in SOFC. However, ceria powders usually possess poor sinterability that sophisticates manufacturing the materials. To improve the sintering of the powder, one can change its morphology or/and use non-conventional methods for sintering, for example, spark plasma sintering (SPS). In this work, 10GDC powders were obtained by thermal decomposition of the plate-like oxalate precursor $\text{Ce}_{1.8}\text{Gd}_{0.2}(\text{C}_2\text{O}_4)_3 \cdot 10\text{H}_2\text{O}$. The synthesis procedure was analogous to that for $\text{Ce}_2(\text{C}_2\text{O}_4)_3 \cdot 10\text{H}_2\text{O}$, described in [1] but with addition 10 mol. % $\text{Gd}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ at the initial stage. This work aimed to investigate the influence of the thermal decomposition conditions on the 10GDC morphology and the properties of the ceramics sintered by SPS. The main factor that influenced the morphology was shown to be water vapor pressure during the dehydration stage (at 120°C). When it was high enough (**Q**uasi-**E**quilibrium conditions),

the crystalline intermediate product formed, and fragmentation of the crystal occurred. Otherwise, the crystal retained its shape and sizes (**p**seudo**m**orph), forming a poorly crystallized dehydrated product. After oxidation of the two dehydrated products in air at 300°C, the powders of 10GDC-pseudo and 10GDC-QE were obtained (Fig.1, left). Peaks' positions in the PXRD pattern of each sample correspond to the fluorite structure (Fm3m). Crystallite sizes calculated from the peaks broadening were about 5-6 nm in each case that correlated with TEM data. However, the sizes and shape of the nanoparticle agglomerates tailored at the dehydration stage were different.

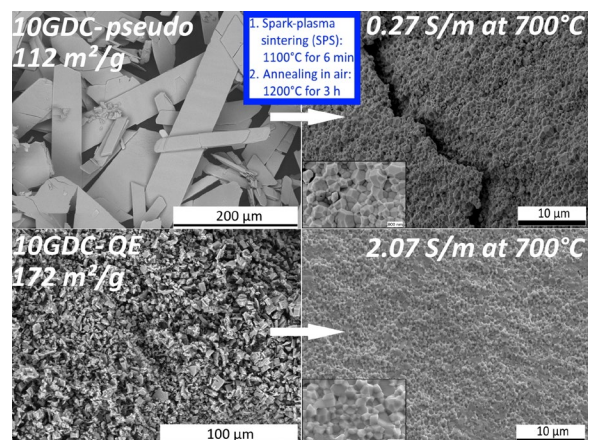


Fig. 1 Morphology of initial 10GDC powders and ceramics obtained by SPS.

The powders of 10GDC were compacted

and sintered using SPS under the same conditions. Then the samples were annealed in air at 1200°C to oxidize samples and to get rid of the rest of carbon. The obtained ceramics were investigated by FEG-SEM (Fig.1, right). Unlike the 10GDC-pseudo sample, the 10GDC-QE sample obtained from the precursor dehydrated under high vapor pressure had no visible fractures. This sample also possessed much better values of ionic conductivity (2.07 S/m at 700°C) and the smaller activation energy (66 kJ/mol) (Fig.2). Thus, dehydration conditions of the chosen precursor were found to be an essential factor controlling morphology of 10GDC powder. Changing the powder morphology was shown to lead to obtaining ceramics with varied characteristics.

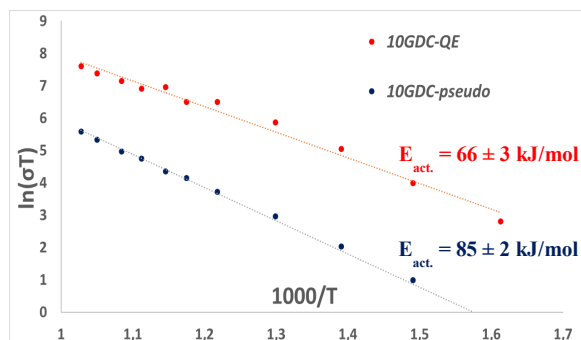


Fig. 2 Ionic conductivity measurements on the 10GDC ceramic samples obtained.

References

- [1] D.V. Maslennikov, A.A. Matvienko, S.A. Chizhik, A.A. Sidelnikov, Synthesis and structural characterization of ceria nanoparticle agglomerates with shape inherited from an oxalate precursor, *Ceramics International*. 45 (2019) 4137–4141.