Spark Plasma Sintering applied on energy materials: illustration with thermoelectric and magnetocaloric applications

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Plateforme IdF de frittage SPS (https://www.icmpe.cnrs.fr/plateformes/frittage-flash-plateforme-regionale/)

The "Spark Plasma Sintering" (SPS – also called Field Assisted Sintering Technology), is a recent densification process allowing to shape, to synthesize and to bond a wide range of materials: metals, ceramics, polymers, composite materials. The sintering powder is put in a graphite (or steel, or tungsten carbide…) die between two conductive electrodes which also transmit a uniaxial pressure. The whole is in a vacuum chamber or under a controlled atmosphere. A very high current (DC) flows through the die and the sample, as successive pulses at a defined frequency, which allows a very quick temperature rise and a complete sintering in a few minutes. This technique enables a good control of the material structure down to nanometric scales especially.

Two cases studies will be presented. The first one being the glass-ceramisation process in the Cu-As-Te system to produce thermoelectric materials[1]. SPS technique has been used to sinter amorphous powder and to shape it through viscous flow mechanisms, as well as to generate glass-ceramic material by controlling nucleation and crystals growth in the glassy matrix. Glass-ceramics containing metastable β-As_2Te_3 crystalline phase have been obtained in the Cu-As-Te system through the SPS experiments from amorphous powder by varying the dwell time at constant pressure and temperature. The effect of ball-milling on the raw powder has also been studied on the glass-ceramisation process as well as on the thermoelectric properties.

The second case study will be focused on reactive spark plasma sintering (R-SPS) to produce perovskite-type substituted lanthanum manganites $La_{1-x}A_xMnO_3$ (A = Na, Ca) for magnetocaloric applications[2]. The manganites were synthesized from raw oxide and/or hydroxide powders in different atomic ratios using the R-SPS technique, which appears to be a new way to make reactive solid state chemistry. Magnetic dense ceramics were elaborated, with specific microstructures different from the one resulting from a classical solid state chemistry route.

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[1] Vaney *et al* J. Mater. Chem. A 2013, 1, 8190; Vaney *et al* Inorg. Chem. 2018, 57, 754; Morin *et al* J Am Ceram Soc. 2019, 102, 2684.

[2] Regaieg *et al* Mater. Letters 2012, 80, 195 ; Regaieg *et al* Mater. Chem. Phys. 2013, 139, 629 ; Regaieg *et al* Mater. Res. Express 2014, 1, 046105; Ayadi *et al* J. Alloys Compds 2018, 759, 52