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SPS for chemical preparations

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Key Words: SPS, mass transport, chemical reaction, solid-state electrochemical reaction.

The Spark Plasma Sintering (SPS) has been developed for shaping materials by combining simultaneously uniaxial pressure (using direct contact with the sample) and the application of an direct electric field. Even though the key mechanisms controlling SPS are still debated, this technique shows a number of advantages – (i) reduction of sintering time, (ii) rapid densification and (iii) grain growth suspension – which are helpful for tailoring the microstructure of functional materials and thus optimize their electronic transport, mechanical and/or thermal properties.
The diffusion rate of SPS conditions was reported to become extremely large at relative low temperatures due to the pulsed DC electric field. The mass transport occurring during the sintering process can be used for performing chemical reactions, especially as an alternative synthesis route for intermetallic and inorganic compounds, of which, some can be obtained only with difficulties by other techniques. The single-step synthesis of species starting from precursors can be provided by the diffusion-controlled reaction at SPS conditions. Several compounds were successfully synthesized, e.g. transition-metal oxides, binary and ternary borides, silicides, germanides [1–4].
Recently, the successful chemical synthesis of the clathrate phases was performed in an SPS setup. They were obtained via a solid-state electrochemical variant of a redox reaction by using binary or ternary alkali-metal Zintl phase's precursors. Clathrate phases form by oxidation of Si^{4-}_4 at the anode while the alkali metal is reduced at the cathode. While the influence of applied current, temperature, alkali metal partial pressure, and relative size of guest and framework cages clearly influence the clathrate formation, a definitive understanding of the exact reaction mechanism is of great interest and is in focus of an ongoing study [5, 6].

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