Effect of process variables on synthesis of MgB₂ by a high energy ball mill

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Abstract. The discovery of superconductivity of MgB₂ in 2001, with a critical temperature of 39 K, offered the promise of important large-scale applications at around 20 K. Except than the other featured synthesis methods, mechanical activation performed by high energy ball mills, as bulk form synthesis or as a first step of wire and thin film productions, has considered as an effective alternative production route in recent years. The process of mechanical activation (MA) starts with mixing the powders in the right proportion and loading the powder mixture into the mill with the grinding media. The milled powder is then consolidated into a bulk shape and heat-treated to obtain desired microstructure and properties. Thus, the important components of the MA process are the raw materials, mill type and process variables. During the MA process, heavy deformation of particles occur. This is manifested by the presence of a variety of crystal defects such as dislocations, vacancies, stacking faults and increased number of particle boundaries. The presence of this defect structure enhances the diffusivity of solute hence the critical currents and magnetic flux pinning ability of MgB₂ are improved. The aim of the present study is to determine the effects of process variables such as ball-to-powder mass ratio, size of balls, milling time, annealing temperature and contribution of process control agent (toluene) on the product size, morphology and conversion level of precursor powders to MgB₂ after subsequent heat treatment. The morphological analyses of the samples were performed by a high vacuum electron microscope ZEISS SUPRA VP 50. The phase compositions of the samples were performed with an Rigaku-Rint 2200 diffractometer, with nickel filtered Cu Ka radiation and conversion level. The MgB₂ phase wt % was calculated by the Rietveld refinement method. The obtained results were discussed according to the process variables to find out their affect on the structure of MgB₂ and to determine the optimum synthesis condition. It was found that the ball-to-powder mass ratio of precursors mixed and the milling time remarkably affect the MgB₂ phase wt % and product size.

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