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Microstructural analysis of magnesium diboride (MgB_2) doped with ZrB_2 and TaB_2 through high energy ball milling and heat treated using HIP

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Abstract – The doping of MgB_2 superconductors with ZrB_2 and TaB_2 diborides represented, even with small amounts of dopants, significant increases in the critical current densities J_c in applied magnetic fields and in the upper critical fields H_{c2}. X-ray diffractograms were obtained for samples doped with 2%, 5% and 10% in weight of dopant. The analysis of the results showed agreement with previous and literature results, and added important data about the mixing of these powders. Through the crystalline structure refinement using the Rietveld method, a decrease of the crystallite sizes could be observed for the compounds with the increase of the milling time. For the MgB_2 the decrease was more pronounced than for the ZrB_2 or TaB_2 dopants.

MgB_2 superconductors can be used in applications only when the critical current densities J_c and critical magnetic field H_{c2} are optimized simultaneously. The best optimization is related to an improvement in grain connectivity and addition of suitable doping [1].

In this work were developed and analyzed MgB_2 samples with addition of other diborides: ZrB_2 and TaB_2. Commercial precursor powder of MgB_2 was mixture to the diborides following the $Mg_{1-x}Ta_xB_2$ relation, where x = 0; 0,02; 0,05; and 0,10, corresponding to the atomic concentration of the doping compound.

The mixture were made in a high energy ball mill. All preparation steps were realized in a controlled atmosphere of nitrogen in a glove-box, in other to reduce the contamination and formation of undesired phases. The powders were compacted using cold isostatic pressing (CIP) under pressures of 220MPa. These pressed samples were placed in stainless steel tubes, evacuated, and heat treated using hot isostatic pressing (HIP) at 1000°C, 24h, 220MPa. This preparation procedure avoided contamination of the samples. The controlled heat treatment promoted high densification of the samples with very good grain connectivity.

The X-ray analysis, Figure 1, showed that the ball milling was efficient to dope the MgB_2, maintaining the hexagonal crystalline structure, and generating efficient pinning centers. The Rietveld analysis helped to determine the crystalline parameters of the mixed material, creating conditions to analyze the doping procedure of the samples. The analysis of the results showed agreement with previous and literature results, and added important data about the mixing of these powders. A decrease of the crystallite sizes could be observed for the compounds with the increase of the milling time. For the MgB_2 the decrease was more pronounced than for the ZrB_2 or TaB_2 dopants. Comparing the milling methods with SPEX and Pulverisette mills, it was observed that the differences found between the results were mainly due to the energetic differences involved in the milling processes, and consequently in doping efficiency. With the Pulverisette, it was possible to observe the almost invariance of the crystallite size of the dopants in relation to the milling time and the expected change on the crystallite size of the magnesium diboride. The milling with SPEX mill was more efficient for the doping process of MgB_2. Figure 2 shows the decrease of the crystallite sizes versus milling time found for the MgB_2+5at.%ZrB_2 samples milled using the Pulverisette mill.

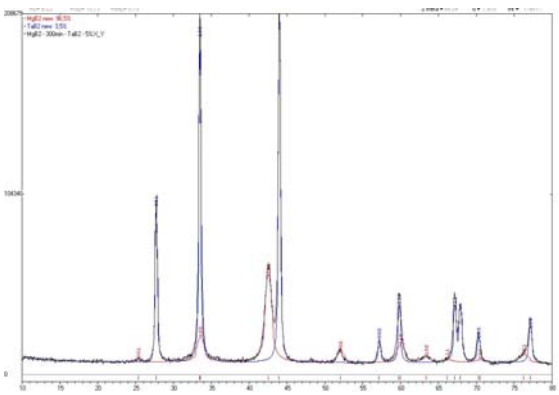

Figure 1: X-ray diffractogram of the MgB_2 + 5at.%TaB_2 heat treated at 1000ºC/24h/220MPa using HIP.

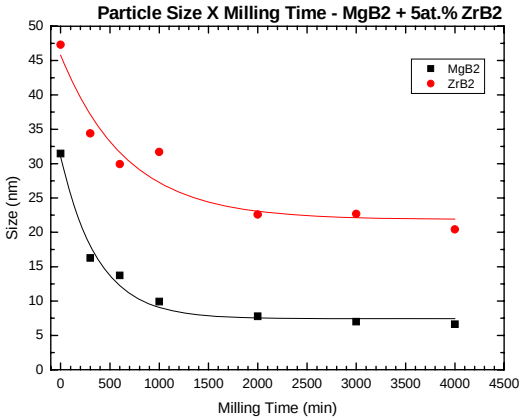

Figure 2: Crystallite size versus milling time for the MgB_2 + 5at.%TaB_2 sample.

[1] D. Rodrigues Jr., B. J. Senkowicz, J. M. Hanson, D. C. Larbalestier, and E. E. Hellstrom, *Adv. Cryog. Eng.* 54 (2008) 359-366.