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Formation of magnesium diboride-based materials with high critical currents and mechanical characteristics by high-pressure synthesis

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Abstract

The developed method of high-pressure synthesis (HPS) allows producing nanostructural highly dense material based on MGB_2, which possesses the highest superconducting and mechanical characteristics among the known world analogues, in the form of blocks that are suitable for application in SC electromotors and pumps. Additions of Zr can increase critical current density (j_c) of synthesized at 2 GPa and 750-800 °C MGB_2 in the same manner as additions of Ta or Ti, i.e. due to the absorption of impurity hydrogen forming the ZrH_2. The formation of ZrB_2 phase at higher synthesis temperatures (about 950 °C) in HPS MGB_2 does not result in the j_c increase. Some increase in j_c of HPS MGB_2 at 10 K in the fields higher than 8 T was observed when nano-SiC was added. The additions of Zr, Ta or Ti can prevent the harmful MgH_2 impurity phase from appearing and hydrogen from being introduced into the material structure. Besides, the presence of additions in HPS MGB_2 promotes the formation of a larger amount of Mg-B (most likely MGB_2) inclusions in the Mg-B-O material "matrix" that in turn leads to the increase of j_c of the material in magnetic fields.

Keywords

Formation, magnesium, diboride, based, materials, high, critical, currents, mechanical, characteristics, high, pressure, synthesis

Disciplines

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Formation of magnesium diboride-based materials with high critical currents and mechanical characteristics by high-pressure synthesis

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Abstract. The developed method of high-pressure synthesis (HPS) allows producing nanostructural highly dense material based on MgB_2, which possesses the highest superconducting and mechanical characteristics among the known world analogues, in the form of blocks that are suitable for application in SC electromotors and pumps. Additions of Zr can increase critical current density (j_c) of synthesized at 2 GPa and 750-800 °C MgB_2 in the same manner as additions of Ta or Ti, i.e. due to the absorption of impurity hydrogen forming the ZrH_2. The formation of ZrB_2 phase at higher synthesis temperatures (about 950 °C) in HPS MgB_2 does not result in the j_c increase. Some increase in j_c of HPS MgB_2 at 10 K in the fields higher than 8 T was observed when nano-SiC was added. The additions of Zr, Ta or Ti can prevent the harmful MgH_2 impurity phase from appearing and hydrogen from being introduced into the material structure. Besides, the presence of additions in HPS MgB_2 promotes the formation of a larger amount of Mg-B (most likely MgB_2) inclusions in the Mg-B-O material "matrix" that in turn leads to the increase of j_c of the material in magnetic fields.

1. Introduction

The structure of MgB_2 high-pressure synthesized from Mg and B, which in accordance with XRD analysis, contains mainly a well-crystallized MgB_2 phase, has turned out to be more complicated as shown by SEM and microprobe examinations [1] (figure 1). In parallel with Mg and B the nanostructure of the main "matrix" phase of the samples contains oxygen (Mg-B-O) and is superconducting. Mg-B (or most likely monocrystalline MgB_2) inclusions of size from 10 μm down to 200 nm or even smaller are distributed throughout the "matrix". Energy-dispersive analysis (figure 1d) has shown that the amount of Mg in the "matrix" with respect to B is much higher than is needed by the MgB_2 stoichiometry, while the stoichiometry of "black" Mg-B inclusions corresponds well to MgB_2. Usually a larger amount of Mg-B inclusions in the structure of HPS MgB_2 corresponds to a higher j_c and irreversibility field, H_{irr}, at 30-10 K [1,2]. Samples, with higher SC characteristics, contain some amount of pure Mg and lesser amount of MgH_2 impurity or this phase is absent at all.

A number of investigations have been performed to study a possibility to produce additional pinning centers in the MgB_2 structure by chemical doping. Promising results have been obtained by adding Ta,Ti, Zr and nano-SiC [1-4]

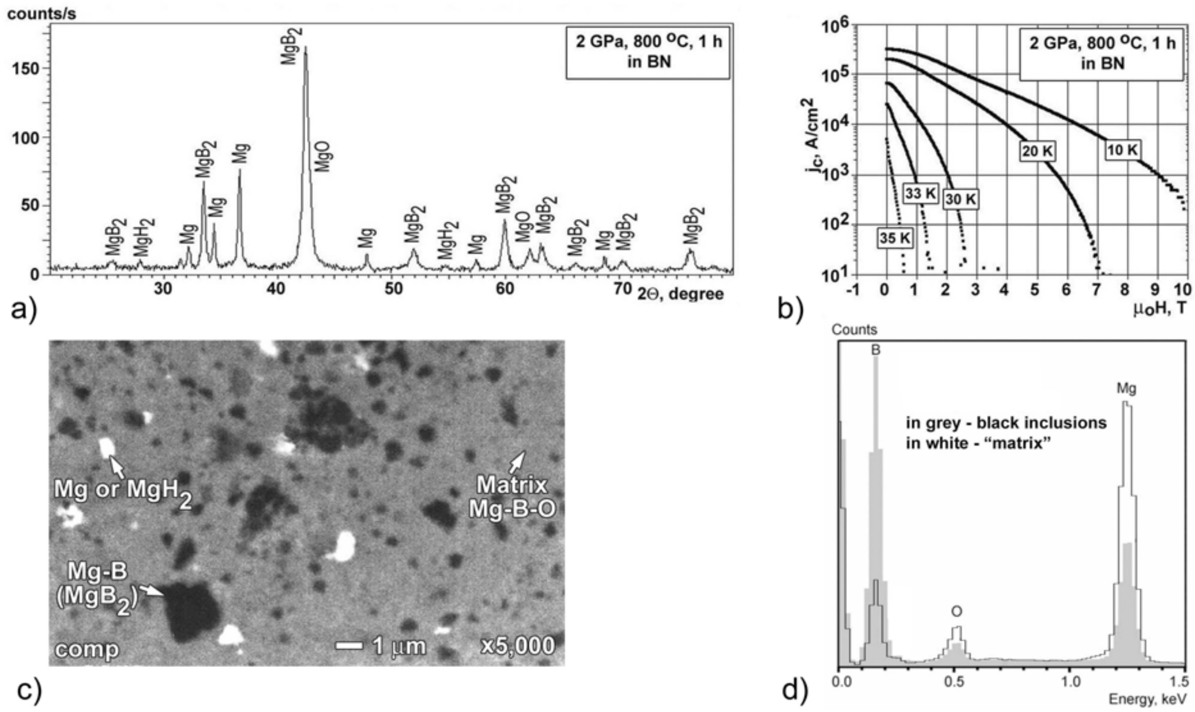

Figure 1. Characteristics of the sample synthesized at 2 GPa, 800 °C for 1 h from Mg and B (without additions): (a) X-ray pattern; (b) critical current densities (j_c) at different temperatures vs. magnetic field (μ_oH) variation; (c) backscattering electron images obtained by SEM; (d) energy-dispersive spectra (gray-colored spectrum is the spectrum of the "black" Mg-B inclusions, white-colored spectrum is the spectrum of the "matrix" Mg-B-O phase of the structure shown in figure 1c).

2. Experimental

Metallic Mg chips and amorphous B (of 1 μm, MaTecK, 95-97% purity), were taken in the stoichiometric ratio of MgB_2. To study the influence of Zr, Ti, Ta, or nano-SiC additions, the Zr (of 2-5 μm, MaTecK, 94-98% purity), Ti (of 1-3 μm, MaTecK, 99% purity), Ta (of 1-3 μm) or nano-SiC (20-30 nm) powders were added to the stoichiometric mixture of Mg and B in amounts of 2 or 10 wt%. Components were mixed and milled in a high-speed activator for 1-3 min. The X-ray study of the initial Mg, Zr, Ti, Ta, SiC and B showed that the materials contained no impurity phases with hydrogen (the accuracy being 3-5%). The high pressure (2 GPa) - high temperature (750-950 °C) conditions for 1 h were created in a recessed-anvil type high-pressure apparatus (HPA) (sample was in contact with hexagonal BN). The structure was studied using SEM and XRD. The j_c was estimated on 3 mm samples using Oxford Instruments 3001 vibrating sample magnetometer (VSM).

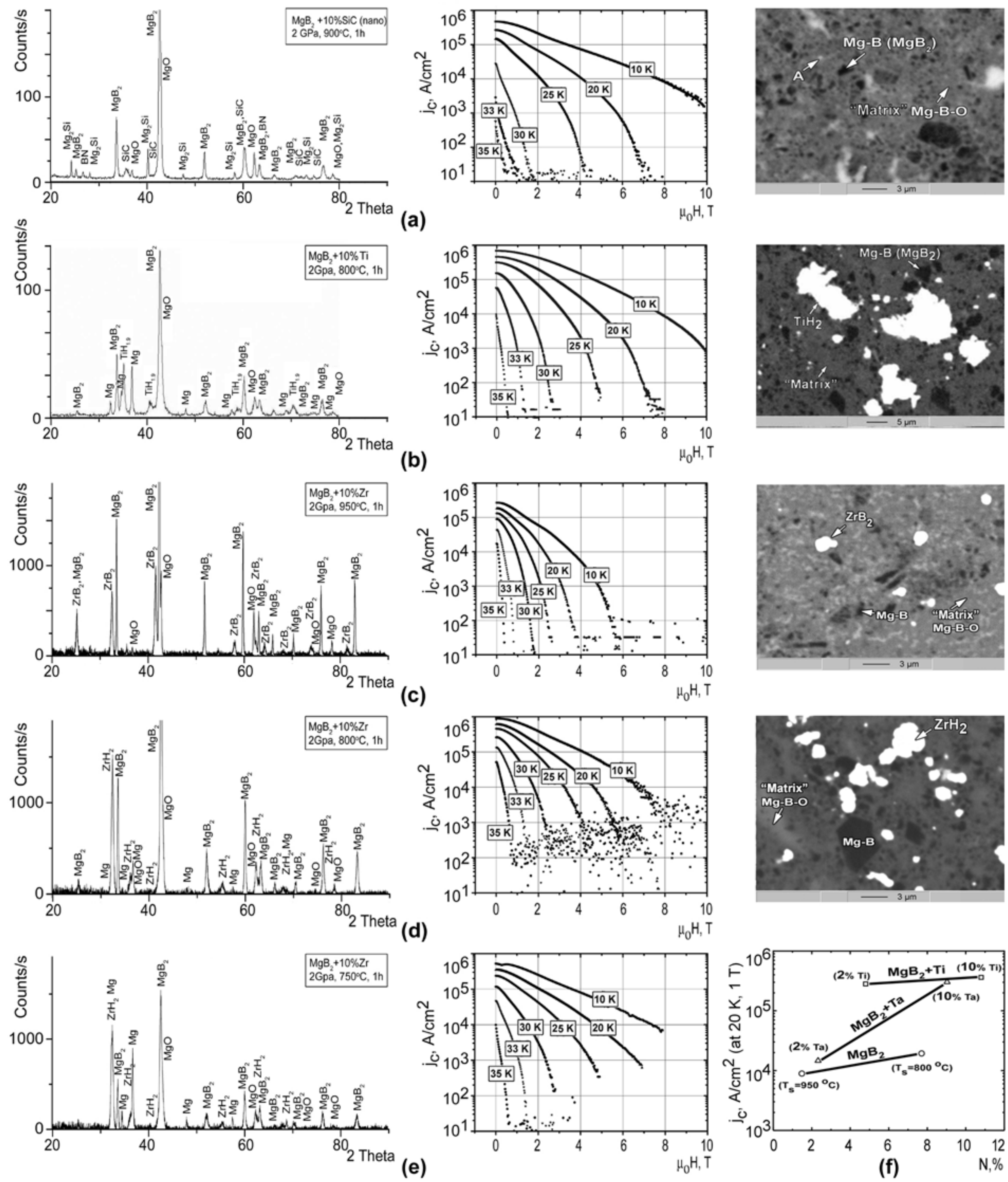

Figure 2. (a-e) X-ray patterns, dependences of *j*c on magnetic fields, μ_oH, and structure obtained by SEM in backscattering electron image of the HPS-MgB_2 with additions of SiC, Ti and Zr. (Regimes of synthesis and amount of additions are given in the pictures). In figure 1a, the letter "A" marks the phase that contains Si C, Mg, B; (f) – the j_c vs. amount of "black" Mg-B inclusions, N, for HPS-MgB_2 samples without additions and with additions of Ta and Ti (N,%, was calculated as a ratio of the area that is occupied by "black" inclusions in the image of the structure obtained at 1600x magnification to the total area of the image obtained by SEM in the backscattering electron regime).

3. Results and discussion

Figures 2 a-e demonstrate the results of the study of HPS MgB_2 with additions of nano-SiC, Ti, Zr and figure 2f shows the results of the quantitative investigation of the amount of “black” inclusions and j_c of the HPS MgB_2 samples without additions and with additions of Ta and Ti. All the additions under study induced an improvement of j_c in HPS MgB_2. The most pronounced improvement of j_c is observed when Ti and Zr are added. The additions of nano-SiC increases the j_c value at 10 K in the fields higher than 8 T. Usually the improvement in critical current density in the case that Ti or Zr are added to the materials synthesized at ambient pressure is explained by the formation of the TiB_2 or ZrB_2 thin layers at grain boundaries that increase the number of pinning centers, which is ascribed to a j_c improvement caused by doping with these elements [4]. The main effect of Ti, Zr and Ta in all cases for HPS MgB_2 can be explained by the absorption of impurity hydrogen (the source of which can be materials of high-pressure cell surrounded the sample during synthesis) to form $TiH_{1.94}$, ZrH_2 or Ta_2H. Additions of Zr, Ti or Ta can prevent the harmful (for j_c) MgH_2 impurity phase from appearing and hydrogen from being introduced into the material structure. The appearance of ZrB_2 (at synthesis temperature T_S=950 °C, figure 2 c) does not affect the j_c of HPS MgB_2 as compared to the j_c of MgB_2 synthesized at the same temperature under the same conditions when Zr was not added (curves for the latter case are shown in [1] in Fig.1a).

A decrease in T_S results in an increase of the amount of Mg-B inclusions and an increase in the amount of Ti or Ta provokes the increase in the amount of these inclusions as well (figure 2 f). The correlation between the amount of Mg-B inclusions and increase of j_c is not so strict because several factors affect the j_c. For example, a decrease in the T_S results in an increase of MgH_2 phase formation (harmful to j_c) and in an increase of the amount of free Mg and Mg-B inclusions that positively affect j_c. Besides, a decrease in the T_S can lead to a decrease in the material density. But many observations allow us to conclude that j_c is most strongly influenced by the amount of Mg-B inclusions.

The highest j_c for HPS MgB_2 with nano-SiC additions was observed at T_S=900 °C. SiC does not absorb hydrogen and at low T_S (750-800 °C) in HPS MgB_2, MgH_2 forms and hydrogen probably enters into the structure of material decreasing the j_c. At higher T_S (900 °C) hydrogen seems to be partly liberated from the pressure cell during the synthesis and the grains “A” containing Si, C, Mg, B or Mg_2Si and SiC found by X-ray (figure 2 a) may serve as pinning centers (instead of Mg-B inclusions whose amount decreases at such T_S). We do not rule out the opinion as to MgB_2 with nano-SiC addition synthesized under ambient pressure that SiC may be incorporated into the MgB_2 lattice and thus facilitate the intragrain pinning.

The hardness of the HPS material (HPS MgB_2 with 10% Ta) measured by a Vickers indenter under a load of 148.8 N is H_v= 10.12±0.2 GPa and the fracture toughness under the same load is K_{1C}=7.6± 2.0 MPa·$m^{0.5}$. The HPS MgB_2 without additions has H_v=16.85±0.74 GPa and K_{1C}=4.24± 0.14 MPa·$m^{0.5}$ under a 4.96 N-load. Using the proposed method, blocks of 32 mm in diameter and up to 20 mm in height and quadratic blocks measuring 28×28×10 mm can be high-pressure synthesized. The HPS MgB_2 material tested in SC motor at 20 K has shown operating characteristics similar to those of MT-YBCO at 20 K.

4. Conclusions

Highly dense alloyed HPS MgB_2 shows j_c at 20 K higher than: 10^5 A/cm^2 up to 3 T, 10^4 A/cm^2 up to 5 T and 10^3 A/cm^2 up to 7 T fields and has high mechanical characteristics.

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