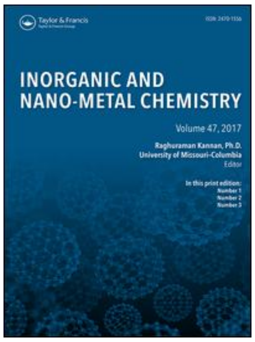

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Synthesis and superconducting properties of the MgB_2@BaO composites

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ABSTRACT
Synthesis of the magnesium diboride doped by microparticles of barium oxide (MgB_2@BaO) composites using a solid-state reaction method has been described. The proposed method gives the possibility to enhance the critical current density of the investigated samples at low temperature in the inert atmosphere. The superconducting parameters (T_c and J_c) have been measured. The influence of the doping agents on superconducting parameters of MgB_2 has been analyzed. Obtained results revealed that the best optimal parameters are for 0.7MgB_2@BaO sample that shows the highest critical transition temperature 39.3 K and critical current density 1.7*10^6 A/cm^2. It was found that an increasing BaO content leads to the formation of a useful phase of BaB_6, which demonstrates positive effects on the superconducting properties, acting as the effective pinning centers.

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Magnesium diboride; barium oxide; combustion; superconductor; doping

Introduction

According to the Bardeen-Cooper-Schrieffer theory,[1] the highest critical transition temperature (Tc) limits in metallic superconductors at ∼30 K. However, such intermetallic superconductors as MgB_2 have changed the previous approaches. Magnesium diboride has been synthesized and investigated in 1953,[2] but its superconducting properties were discovered by Japanese scientists only in 2001.[3] Its Tc is 39 K that is the highest among phonon-mediated superconductors. For the synthesis of the MgB_2 several routes are used, such as high-temperature solid-state reaction; the powder-in-tube *ex-situ* and *in-situ* modes; infiltration and growth processing method; hybrid physical-chemical vapor deposition.[3] Extensive research has been undertaken to improve the flux pinning in MgB_2, which could enhance its electromagnetic performance.[4] Nowadays, the techniques used to improve critical current density (Jc) and the flux pinning in MgB_2 include chemical doping, irradiation and thermomechanical processing.[4,5] Such processes as hot isostatic pressure (HIP), cold drawing (CD), cold rolling (CR), cold pressure (CP), and doping have been shown to improve these properties for MgB_2. HIP process increases density and homogeneity of MgB_2, number of connections between grains, and dislocation density while it reduces the size of voids.[6,7,8] It has been showed that CR also improves the homogeneity of MgB_2 material and leads to an increase *in-situ* MgB_2 density of ∼37% and *ex-situ* MgB_2 density – 19%.[9–11] The CD process leads to the rising of the length and outer surface of MgB_2 grains, reducing their thickness and improving Jc wires.[12,13]

To increase the values of the two parameters T_c and J_c, the introduction of controlled amounts of doping agents is widely used.[14] Different types of pinning centers can be introduced, e.g., grain boundaries, point defects as well as impurities and lattice variations brought on by doping.[15–20] In order to make the formation of pinning centers that could effectively increase of J_c, they should exhibit sizes as large as the coherence length that in the MgB_2 is in the range of 2-10 nm. Chemical doping also influences on T_c of MgB_2. Some additives lead to the decrease of T_c and the loss of superconductivity, such as doping by Al, Li. Other elements, such as Be, don’t dope the MgB_2 in the lattice and does not affect T_c at all. However, some dopants may work as pinning centers if a proper microstructure of the second phase can be formed in MgB_2.[21] For example, the authors [22,23] studied the effect of Y_2O_3 addition on the superconducting properties of bulk MgB_2 and could improve the critical current density of bulk MgB_2 sample at high field without decreasing of superconducting transition temperature due to the forming of useful phase YB_4 in MgB_2 sample. Also, carbon doping is considered as one of the most effective ways to improve the J_c of superconductors based on MgB_2, especially at high field.[24–27]

Besides the unusually high T_c, MgB_2 has a large coherence length, low anisotropy, and transparent grain boundaries. The most important difference between MgB_2 and other practical superconductors is that it has two superconducting gaps originating from two different bands. Tuning the scattering rates between the two bands improves the superconducting properties and the practical applicability of

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MgB_2. The different methods of fabrication of MgB_2 conductors are described and compared. Fabrication of long length MgB_2 conductors is relatively easy and non-expensive method that allows the use of a variety of sheath materials with suitable barriers or reinforcement. The conductors have much better mechanical properties for practical applications. The critical issues and the challenges are to be addressed for realization of MgB_2 superconductors as the first choice for high field magnet applications. At present MgB_2 is most suited for 20–25 K operation in fields of 1–2 T. However, some investigation indicates that oxide additions based on the rare earth or metalloid elements show improvement of the MgB_2 J_c and the irreversible magnetic field (H_{irr}) without significantly affecting the $T_{c.}$[28] It was found, that the characteristics of the additions and the technological approaches show a strong influence in controlling superconducting properties. For example, dense bulk samples (relative density of 88-99%) of MgB_2 with $Ge_2C_6H_{10}O_7$ as dopants were obtained by *ex-situ* spark plasma sintering.[29] The J_c of the elaborated samples was improved at high magnetic fields. The optimum composition was found for MgB_2 $(Ge_2C_6H_{10}O_7)_{0.0014}$, where J_c (20 K) is 10^2 A/cm^2 at 5.8 T, versus 3.9 T for the reference sample.[30] However, there is no evidence for J_c improving.

In this work, we proposed to use the self-propagating high-temperature synthesis (SHS) technique that allows doping MgB_2 by additives spontaneously due to the exothermic heat of reaction. We used the cheap and widely distributed BaO microparticles as a dopant during the synthesis of the MgB2@BaO superconductor by SHS under high gas pressure. This work aims to identify the effect of doping on superconducting parameters of MgB_2 enhancing its Jc.

Materials and methods

The solid-state technique has been used to obtain MgB_2@BaO samples. The samples were prepared as follow: magnesium (100-200 μm), boron (1-5 μm) powders and BaO (50 μm) in the composition Mg 55.3 wt% + B 44.7 wt% + X_{BaO} (where X = 0.3, 0.7, 1 and 5 wt. %; denote as 0.3MgB_2@BaO, 0.7MgB_2@BaO, 1.0MgB_2@BaO, 5.0MgB_2@BaO respectively) have been ignited under Ar at 2.5 MPa in a high pressure chamber (Figure 1).[27,31] The mixture of the starting materials was loaded into a cylindrical mold and compacted in the form of a pellet at a pressure of 570 MPa to obtain dense material. Self-sustainable combustion was initiated at temperature T = 327 – 377 K. After combustion, the samples were structurally characterized by X-ray diffraction (XRD) using Dron – 4 diffractometer (operating with a Cu-Kα radiation source) and Scanning electron microscope Quanta 200i 3 D. The measurements were carried out on a Physical Property Measurement System of Quantum Design.

The J_c [A/cm^2] parameter was calculated according to the formula $J_c = 30*\Delta M/d$ (Bean's Formula), where ΔM – the difference between the bottom and top of the magnetization of the magnetic hysteresis, d – the average grain size. The real density of samples was measured by the Archimedes principle. Current work is devoted to the "pilot" experiments; therefore, all experiments were carried out in 100 Oe.

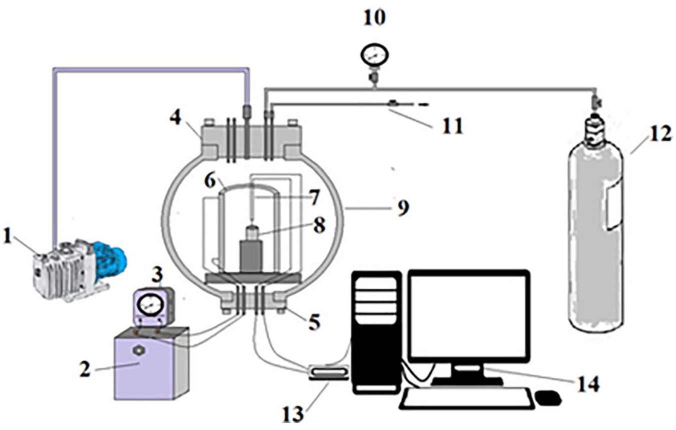

Figure 1. Synthesis of the MgB2@BaO samples: 1 – vacuum pump; 2 – transformer; 3 – ammeter; 4 – the top cover of the reactor; 5 – the bottom cover of the reactor; 6 – tubular heating furnace; 7 – thermocouple; 8 – sample; 9 – the case of the reactor; 10 – gauge; 11 – intake and exhaust valves; 12 – nitrogen cylinder; 13 – the data acquisition system LTR-U-1, 14 – computer.[25,26]

Table 1. MgB_2@BaOcomposition (wt%) according to the XRD data.

Sample	MgB_2	MgB_4	MgO	Mg	BaB_2	BaB_6
MgB_2	94.2	–	5.8	–	–	–
0.3MgB_2@BaO	85.6	3.2	6.8	1.9	2.1	0.3
0.7MgB_2@BaO	91.7	2.3	5.4	–	–	0.7
1.0MgB_2@BaO	88.4	2.2	7.7	–	–	1.7
5.0MgB_2@BaO	88.1	1.9	8.1	–	–	2.0

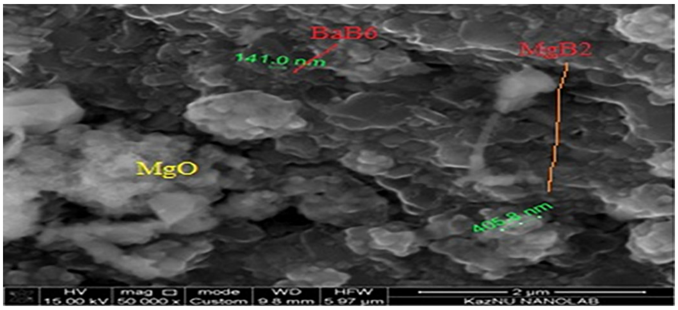

Figure 2. Scanning electron microscope image of 0.7MgB_2@BaO sample.

Results

As is known, during SHS synthesis incorporated BaO particles interact with Mg that leads to the generation of the new boron-containing phases. This statement confirms obtained XRD patterns of elaborated samples (Table 1). Clearly, all the peaks inherent to MgB_2 in all samples and some MgO, MgB_4, BaB_4, BaB_6, as well as unreacted Mg impurity phases, were detected. According to the SEM analysis (Figure 2), the average grain sizes of the samples are ranging from 140 to 400 nm. It should be noted that MgO phase is always forming during the synthesis of superconductors based on MgB_2 by any technique. This indicates that most MgO phase may be coming from the starting material. Therefore, it is crucial to use high purity initial components. Also, it is well known that decreasing the MgO phase leads to the improvement of the Jc value and different techniques were explored for the obtaining of such effect.[32–34] Our results are in good agreement with the previous ones.

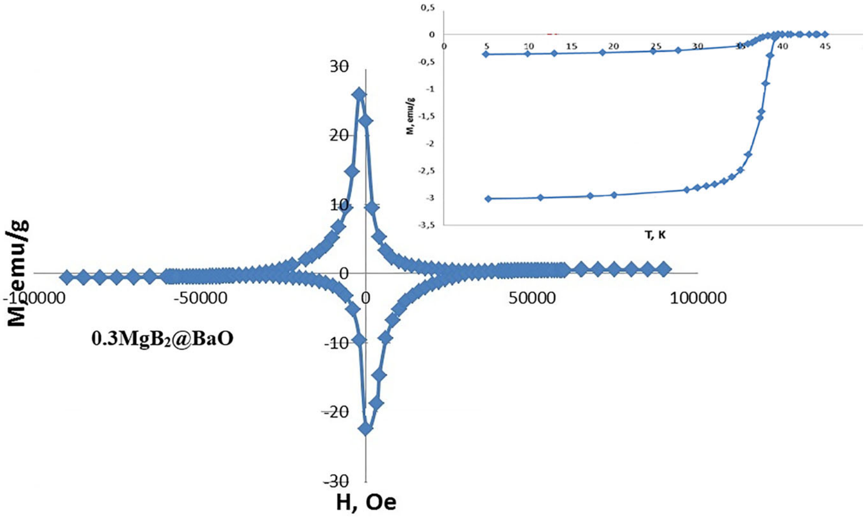

Figure 3. Magnetic hysteresis at 5K and temperature dependence of the magnetization for the system 0.3MgB_2@BaO.

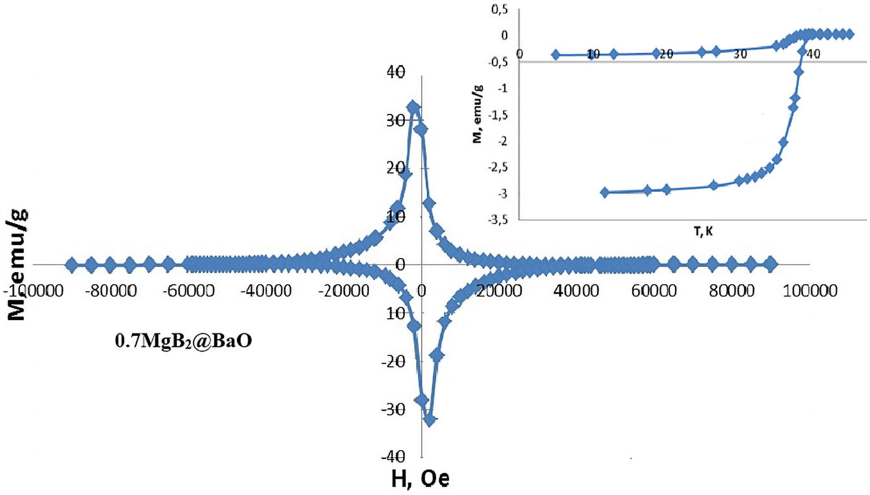

Figure 4. Magnetic hysteresis at 5K and temperature dependence of the magnetization for the system 0.7MgB_2@BaO.

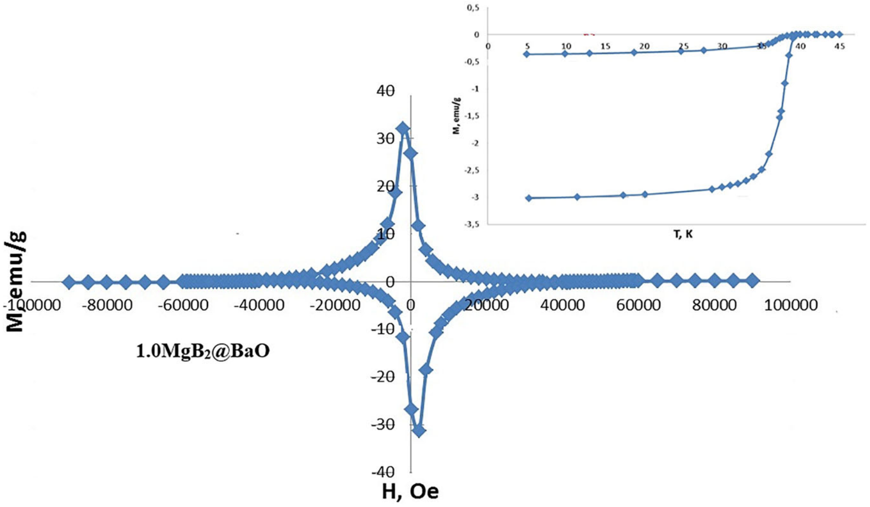

Figure 5. Magnetic hysteresis at 5 K and temperature dependence of the magnetization for the system 1.0MgB2@BaO.

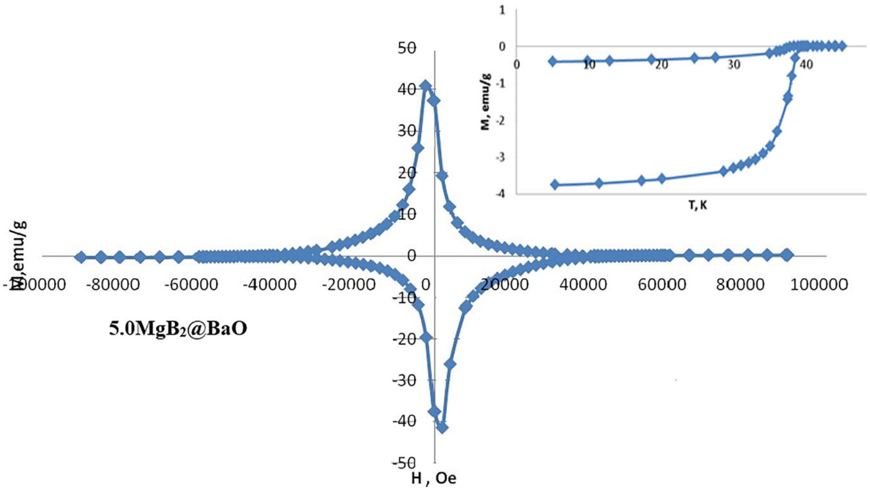

Figure 6. Magnetic hysteresis at 5 K and temperature dependence of the magnetization for the system $5.0MgB_2@BaO$.

Table 2. The calculated values of T_c and J_c for the systems $MgB_2@BaO$.

Name	Critical temperature, T_c (K)	Critical current density J_c (A/cm^2) at 5 K, H = 100 Oe
MgB_2	39.1	0.21×10^6
$0.3MgB_2@BaO$	39.2	1.3×10^6
$0.7MgB_2@BaO$	**39.3**	**1.7×10^6**
$1.0MgB_2@BaO$	39.3	1.3×10^6
$5.0MgB_2@BaO$	39.3	1.1×10^5

The superconducting characteristics of $MgB_2@BaO$ samples were determined by magnetometric measurements. All samples demonstrate a decrease in the magnetic moment at temperature in the range $38\,K < T < 50\,K$, which is preceded by a sharp response of the magnetic moment in the diamagnetic state at $T \sim 39.1\,K$ (Figures 3–6). The calculated J_c is presented in Table 2. The best result has been obtained for the system of $0.7MgB_2@BaO$ – T_c is 39.3 K and J_c is 1.7×10^6 A/cm^2.

It is well known, that the SHS method has several advantages, such as very high reaction rates and low external temperature (due to self-generation of heat). However, its major limitation is the high porosity of combustion products. Therefore, we used external pressure of Ar gas, which allowed us to keep all necessary components (gaseous atoms of magnesium at high T) in the reaction zone during the synthesis formation of MgB_2 phase. We could synthesize a dense sample of $MgB_2@BaO$ is about $2.1\,g/cm^3$ which is about 90% of the theoretical density ($2.33\,g/cm^3$), measured by the Archimedes principle. The denser material demonstrates higher J_c, because of the small amount of the cavities that could act as barriers to flow electrical charge. The presence of secondary phase BaB_6 (an effective pinning centers) and the higher density of material allow us to obtain the second types superconductor with high J_c and T_c.

Conclusion

The positive effect of the pressure of Ar and doping with BaO during the solid-state synthesis of MgB_2 powders on its critical current density J_c has been confirmed. It was found that MgB_2 phase responsible for the increase of critical current. Simultaneously, an increase in the content of barium oxide in the initial mixture leads to the formation of a useful phase of BaB_6, which in turn positively affects the superconducting properties of the composite based on magnesium diboride, acting as effective pinning centers. It is obviously the content of barium oxide significantly influence on the crystallite size (Table 2) in the final product of the synthesis where the grain size of all samples decreases monotonically. It is most likely, that BaB6 impurity plays a key role in enhancing of Jc parameter of MgB2 samples. Simultaneously, this question needs further investigations (as theoretical as well as empirical measurements), especially how the BaB6 phase affects the nature of grain connectivity of the sintered MgB2 sample.

The best optimal result obtained to the system $0.7MgB_2@BaO$ was T_c 39.3 K and J_c 1.7×10^6 A/cm^2, due to the formation of BaB_6 phase in enough content and size. The value of the critical current density of the doped samples is higher than the undoped ones. The easy and quick creation of the material, together with the low cost of production, makes the method very promising for future engineering applications.

Declaration of interest statement

We have no conflict of interest to declare.

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