

EFFECT GRAPHITE ON MAGNESIUM DIBORIDE SUPERCONDUCTIVITY SYNTHESIZED BY COMBUSTION METHOD UNDER ARGON PRESSURE: PART II

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Preliminary Note – Prethodno priopćenje

There are many various techniques to synthesize the superconductors based on magnesium diboride. In this paper, we attempted to obtain a superconducting magnesium diboride in a way by using a High-Temperature Centrifuge and investigated how a centrifugal force can be effect on superconducting parameters of MgB₂ during solid-state combustion. It was the first superconductor based on magnesium diboride synthesized under the influence of centrifugal force in the high temperature centrifuge during solid phase process. As a result of this research, we could determine that a centrifugal force do not impact considerably on the critical transition temperature of the MgB₂ samples (stay at around 37,5 – 38 K). However, it was found that the value of a centrifugal force could effects on the critical current density (J_c) of the samples.

Keywords: magnesium diboride, graphite microparticle, solid-state synthesis, superconductivity, temperature

INTRODUCTION

To date, one of the simplest and most effective methods for the synthesis of magnesium diboride is solid state reaction (SSR). SSR at high pressures is one of the advanced methods for forming a high-density structures for the various materials [1]. A huge advantage of this method is the simplicity and possibility of obtaining fine particle sized materials [2]. Due to the use of external pressure, it is possible to significantly suppress the undesirable process like the evaporation of magnesium during the synthesis and obtain a material with excellent functional characteristics. We have already successfully synthesized the superconductors based on magnesium diboride with different doping agents [3-4]. An undoped magnesium diboride is not so suitable for applying due to the weak flux pinning and low values of critical current density (J_c) and critical transition temperature (T_c). A considerable rise of critical current density in magnesium diboride might be reached via chemical doping with different sorts of material.

Chemical doping is a simple and easily scalable method [5]. An essential increase of critical current density in magnesium diboride can be reached via chemical doping with carbon (C) containing compounds or composites, like B₄C, SiC, C or carbon single-walled nanotubes (CSWN), carbohydrates, hydrocarbon etc. A several research groups synthesized and characterized materials with such chemical formulas as (Mg_{1-z}T_z)B₂

or Mg(B_{1-y}M_y)₂. The agenda was multifaceted: to seek changes in T_c, to conduct tests of superconducting mechanisms in magnesium diboride, and to introduce additional pinning centers, which could lead to a higher critical current density [6]. To replace the boron site, a number of attempts were made with various elements. In most of these attempts, elemental magnesium, boron, and carbon were used as starting materials, and the synthesis was carried out at various pressures and temperatures. One of the difficulties associated with doping magnesium diboride may be the fact that the MgB₂ structure is strong and requires a close atomic level; to achieve substitution, mixing of the dopant with the doped element is required before or during the synthesis [7]. Bean [8] presented details of the synthesis of the optimization and physical properties of almost single-phase carbon-doped MgB₂ with a nominal stoichiometry of Mg (B_{0.8}C_{0.2})₂, synthesized from magnesium and boron carbide (B₄C) as starting materials. The superconducting transition temperature was about at 22 K (more than 17 K lower than in an undoped magnesium diboride). Chunks of magnesium (99,9 % purity) and B₄C powder (99 % purity) were sealed in tantalum tubes, sealed in quartz, and placed in a heated box furnace (heating for 2 hours at 600 °C, and then another 2 hours at 700 °C) and then (after the desired synthesis time) was quenched to room temperature.

EXPERIMENTAL PROCEDURE

The main component of this installation is a metal thick-walled spherical housing, with a wall thickness of 60 mm and a capacity of 45 liters (see Figure 1). Re-

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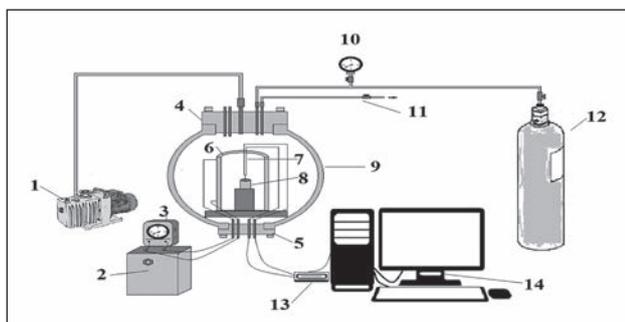


Figure 1 High Pressure Chamber

- 1 a vacuum pump, 2 a transformer, 3 an ammeter, 4 an upper chamber cover, 5 a lower chamber cover, 6 a tube heating furnace, 7 a thermocouple, 8 a sample, 9 a chamber vessel, 10 a pressure gauge, 11 an inlet and outlet valves, 12 an argon baloon, 13 LTR-U-1 data acquisition system unit, 14 a computer

Table 1 The composition of the initial charge

Name of the sample	The composition of the masses wt. / %
Undoped MgB_2	55,3 wt. % Mg+44,7 wt. % B
MgB_2 @ 1 % C	(55,3 wt. % Mg+44,7 wt. % B)+1 wt. % C
MgB_2 @ 3 % C	(55,3 wt. % Mg+44,7 wt. % B)+3 wt. % C
MgB_2 @ 5 % C	(55,3 wt. % Mg+44,7 wt. % B)+5 wt. % C
MgB_2 @ 7 % C	(55,3 wt. % Mg+44,7 wt. % B)+7 wt. % C

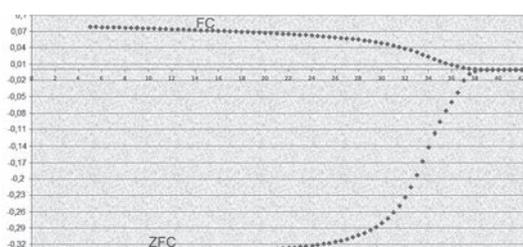
sidual air is removed from the chamber using a vacuum pump. A computer temperature recording unit was used for controlling the measurement of temperature curves during synthesis.

The composition of powder mixtures is shown in Table 1, where, a non-stoichiometric mixture (excess of magnesium) was prepared to compensate for possible losses of magnesium during synthesis due to its high volatility at high temperature. We applied argon pressure to hold most gaseous magnesium atoms in reaction zone during solid-state synthesis where the temperature reached up to 1 200 °C (a boiling point of magnesium is 1 091 °C).

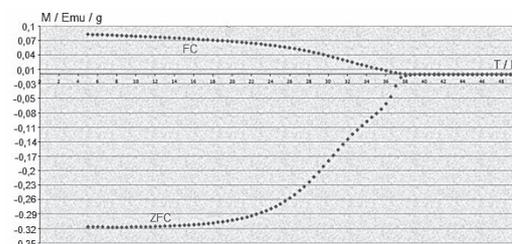
Experimental samples (a height of 20 mm and a diameter of 30 mm) were made by pressing with an effort of 0,4 GPa in order to obtain dense samples. Then, samples placed in the heating furnace of a high-pressure chamber at argon pressure of 2,5 MPa, which value was identified experimentally in our previous works [3]. When temperature reached about 600 – 650 °C in a furnace, a self-ignition occurred and solid phase synthesis started. Combustion temperature during exothermic reaction were in the range of 1 100 - 1 200 °C.

RESULTS AND DISCUSSION

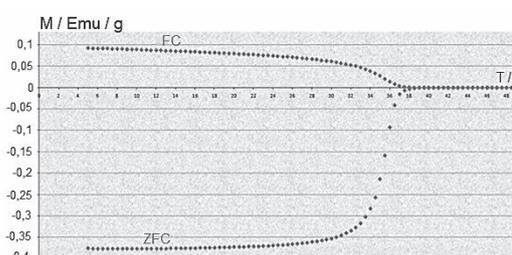
The superconducting characteristics of the samples were measured using magnetometric measurements (Quantum Design PPMS EverCool-II) in the temperature range of 5 - 100 K and magnetic fields of 10 Oe. The designation (ZFC) Zero Field Cooled is a measurement mode when a constant magnetic field is applied in a relatively low temperature region with an increase in its value above the characteristic value. Field Cooled (FC) is a measurement mode when a constant magnetic



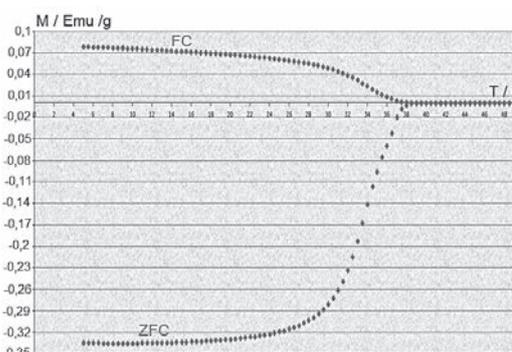
(a)



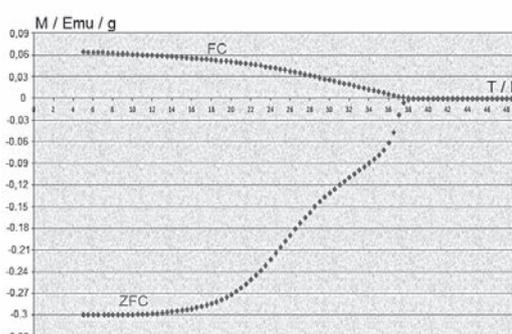
(b)



(c)



(d)



(e)

Figure 2 Temperature dependence of changes in the magnitude of magnetic moments of the undoped and doped MgB_2 . Doping level: a – undoped sample, b - 1 wt. / %, c-3 wt. / %, d -5 wt. / %, e-7 wt. / %

field is applied in a relatively high temperature region with its value falling below the characteristic value. ZFC - pre-cooling of samples to $T < T_C$ (in our case to

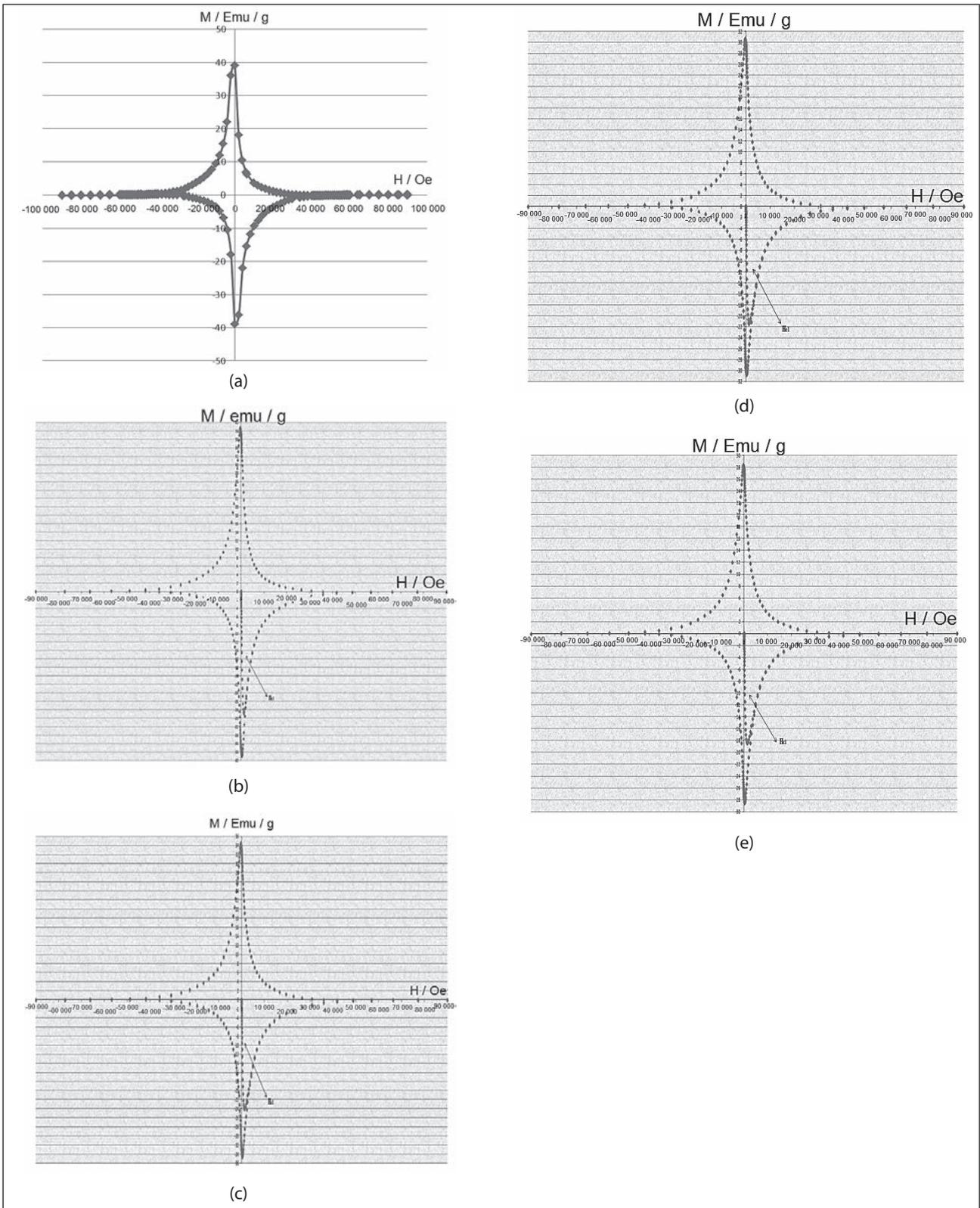


Figure 3 The hysteresis curves for the Mg-B-C system. Doping level: a – undoped sample, b - 1 wt. / %, c-3 wt. / %, d -5 wt. / %, e-7 wt. / %

$T = 4,2 \text{ K}$) in the absence of a field, and FC-at $N \neq 0$. Figure 8 illustrates the temperature dependences of the magnetization $M(T)$ of samples of the Mg-B-C system, measured in the 10 Oe field in the ZFC and FC modes. In this case, the sample is cooled in a zero external magnetic field from room temperature to 5 K, after which a small magnetic field is applied. Then, in this constant

field, the magnetic moment (ZFC) of the sample is measured as the temperature increases. After that, the sample was cooled down again up to 5 K in the same magnetic field (FC).

When measuring the temperature dependences on the magnetization of samples of the Mg – B – C system, a fall in the magnetic moment of samples at tempera-

Table 2 Superconducting characteristics of undoped and doped samples of MgB₂

Name	The critical transition temperature / K	The critical current density (at 5K) / A / cm ²
MgB ₂ @1 % C	38,7	2,1x10 ⁶
Undoped MgB ₂	39,0	2,6x10 ⁶
MgB ₂ @3 % C	38,8	2,9x10 ⁶
MgB ₂ @5 % C	38,5	2,2x10 ⁶
MgB ₂ @7 % C	38,5	2,0x10 ⁶

tures of $38 < T < 40$ K was found, which precedes a straight response of the magnetic moment to the diamagnetic state at $T = 38,5$ K. This temperature characterizes the transition temperature from the normal state to the superconducting one. The obtained data indicate the origin of a superconducting phase in samples of the Mg-B and Mg-B-C systems, at temperatures of more than 38,5 K. The critical current density value of samples was computed by using the Bean's formula [8]:

$$J_c = 30 * \Delta M / d$$

J_c is a parameter of the critical current density of the sample, and d is the average particle size. ΔM is the difference in decreasing and increasing the magnetization curve. The value of the ΔM value was taken from the hysteresis loop curves (see Figures 2-3).

Using this formula, we found the value of the critical current density of magnesium diboride containing graphite microparticles at various concentrations in their own magnetic field. It was found that an increase in the graphite content in magnesium diboride proportionally reduces the value of J_c of the samples. One reason might be that an increase in the content of graphite in the composition distorts the crystal structure of the superconducting phase MgB₂, because the carbon atom radius ($r_c = 0,772$ Å) is much less than the boron atom radius ($r_b = 0,822$ Å). It is determined that doping with 3 wt. / % of micrographite is the optimal dose for obtaining higher critical current densities for MgB₂ samples with the critical current density was $2,9 \times 10^6$. In comparison with work [8], where authors could get a nanographite doped (0,1 wt. / %) magnesium diboride with the following superconducting parameters of the critical current density $1,1 \times 10^6$ at 5 K. This is interesting result, which shows micro particles have more effect on J_c parameter of MgB₂ superconductor than nanoparticles one. Meanwhile, it obvious that doping of micrographites do not have any impact on the critical tran-

sition temperature of samples. Table 2 shows the main superconducting characteristics of graphite-doped magnesium diboride.

CONCLUSION

A superconductor based magnesium diboride with a different content of graphite microparticles was obtained by combustion mode at an argon environment. The offered method allowed the possibility to considerably increase the critical current density of the studied samples at relatively low synthesis temperature in the inert environment. Obtained results revealed that the best optimal parameters are for MgB₂@3 % C sample that shows the highest critical transition temperature 38,8 K and a critical current density $2,9 \times 10^6$ A / cm².

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Note: The responsible for England language is L. D. Sergeeva, Almaty, Kazakhstan.