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The Effect of Inhomogeneous Phase on the Critical Temperature of Smart Meta-superconductor MgB₂

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Abstract

The critical temperature (T_C) of MgB₂, one of the key factors limiting its application, is highly desired to be improved. On the basis of the meta-material structure, we prepared a smart meta-superconductor structure consisting of MgB₂ micro-particles and inhomogeneous phases by an ex situ process. The effect of inhomogeneous phase on the T_C of smart meta-superconductor MgB₂ was investigated. Results showed that the onset temperature (T_C^{on}) of doping samples was lower than those of pure MgB₂. However, the offset temperature (T_C^{off}) of the sample doped with Y₂O₃:Eu³⁺ nanosheets with a thickness of 2 ~ 3 nm which is much less than the coherence length of MgB₂ is 1.2 K higher than that of pure MgB₂. The effect of the applied electric field on the T_C of the sample was also studied. Results indicated that with the increase of current, T_C^{on} is slightly increased in the samples doping with different inhomogeneous phases. With increasing current, the T_C^{off} of the samples doped with nonluminous inhomogeneous phases was decreased. However, the T_C^{off} of the luminescent inhomogeneous phase doping samples increased and then decreased with increasing current.

Keywords Smart meta-superconductor $MgB_2 \cdot Y_2O_3$: Eu³⁺ nanosheets \cdot Inhomogeneous phase \cdot Applied electric field T_C

1 Introduction

Since Akimitsu et al. [1] discovered the binary compound MgB₂ superconductor in 2001, this material has attracted considerable attention because of its simple structure, low cost, large coherence length, and relatively high transition temperature ($T_C = 39$ K) [2]. Improving the superconducting transition temperature of MgB₂ can not only increase its application but also promote the development of superconductivity theory. The most commonly used method in improving the critical temperature (T_C) of MgB₂ is chemical doping. Substituting Mg and B with Al and C, respectively, in MgB₂ forms the displacement doping. However, results showed that the two kinds of doping reduce the T_C of MgB₂ [3–7]. In addition, another possible method for improving the T_C is increasing the density of holes by partially substituting Mg with Li. Nevertheless, experimental results

⊠ Xiaopeng Zhao xpzhao@nwpu.edu.cn showed that T_C is still reduced [8, 9]. The T_C of MgB₂ is reduced because of the presence of the dopant as an impurity in MgB₂, which results in poor grain connectivity and doping into other substances to distort the MgB₂ lattice. Although it is important to increase the T_C of MgB₂ beyond the theoretical value, it also presents considerable challenge and requires further investigation.

Meta-material, a type of artificially structured composite material, is composed of the matrix material and its unit material. The properties of these materials mainly depend on the artificial structure, which can realize many special functions [10–15]. With the development of meta-material, the use of meta-material concept to design superconducting materials to improve its $T_{\rm C}$ has been recognized. We proposed the introduction of electroluminescence (EL) materials in meta-materials to enhance the superconducting transition temperature through EL [16, 17]. Zhang et al. [18] used an in situ solid-phase sintering process to dope Y_2O_3 :Eu³⁺ EL materials in MgB₂; their results showed that the $T_{\rm C}$ of MgB₂ can be enhanced by doping of Y₂O₃:Eu³⁺ EL materials. However, in the in situ sintering process, the raw material B reacts with the Y₂O₃:Eu³⁺ EL material to form the impurity phase of YB₄. To avoid the formation of the impurity phase of YB₄, Tao et al. [19] doped Y_2O_3 :Eu³⁺

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EL material into MgB₂ by an ex situ solid-phase sintering process. Experimental results indicated that the Y_2O_3 :Eu³⁺ EL material doping can improve the superconducting transition temperature of MgB₂; moreover, the morphology and size of the Y_2O_3 :Eu³⁺ EL material affect the T_C of MgB₂. To improve the distribution and connectivity of the inhomogeneous-phase dopants, the effect of different concentrations and sizes of YVO₄:Eu³⁺ microsheets EL material on the superconducting transition temperature of MgB₂ was studied. Results showed that the T_C^{off} of doped samples increases by 1.6 K compared with that of pure MgB₂ when the doping concentration is 2.0 wt%. Recently, Smolyaninov et al. [20–24] proposed that material can be designed as a meta-material structure with an effective dielectric constant $\varepsilon_{\rm eff} \approx 0$, which can improve the $T_{\rm C}$ of the material. They also confirmed the material performance in their experiment.

For superconducting samples, the magnitude of the test current directly affects the accuracy of the test results. Considerably low current tends to decrease the useful voltage signal of the sample. Hence, the requirement of the voltage drop measurement instrument is high. When the current is significantly large, although the requirement of the voltage drop measurement instrument can be reduced, it will increase the thermal effect of the sample. Consequently, a large temperature hysteresis occurs, which affects the acquisition of real data [25, 26]. Ye et al. [27, 28] found that superconducting transition in unconventional superconductors ZrNCl and MoS₂ through carrier doping was induced by an applied electric field. Changing the applied electric field can obtain different transition temperatures; ZrNCl and MoS₂ display a transition temperature $(T_{\rm C})$ of 15.2 and 10.8 K, respectively, on the optimum carrier doping.

On the basis of the idea of meta-material structure, our group proposed a smart meta-superconductor with a sandwich structure, where MgB₂ particles are used as the matrix material; the EL material distributed around the MgB₂ particles are as inhomogeneous phase. When evaluating the curve of the temperature dependence of resistivity (R-T)of the samples, it was found that in the local electric field, MgB₂ particles act as microelectrodes, which promote the EL of inhomogeneous phase EL materials, and that the inhomogeneous phase significantly improves the $T_{\rm C}$ [18, 19]. In this paper, the responses of the critical temperature of MgB₂ to inhomogeneous phase doping and changing the applied electric field are systematically studied. At first, we prepared the Y₂O₃:Eu³⁺, Y₂O₃, and Y₂O₃:Sm³⁺ nanosheet inhomogeneous phases; they were doped into MgB₂ by an ex situ process. The effects of doping Y_2O_3 :Sm³⁺/ Y_2O_3 nonluminous inhomogeneous phase or Y_2O_3 :Eu³⁺ EL inhomogeneous phase on the superconducting properties of MgB₂ were investigated. Then, on the basis of the theory that the material with a meta-structure and an effective dielectric constant close to zero can improve the $T_{\rm C}$ of the material, Y_2O_3 :Eu³⁺ microsheets and nano-Ag solution were doped into MgB₂ to change the dielectric constant of the system; the superconducting properties of doping samples were also evaluated. Finally, we further examined the influence of the applied electric field on the EL and nonluminous inhomogeneous phase doping samples.

2 Experiment

2.1 Preparation of Nanosheets/Microsheets

At first, a certain amount of Y2O3 and Eu2O3 powder were added to 4 mL of concentrated nitric acid under stirring and heating at 70 °C for 1 h to obtain the Y(Eu)(NO₃)₃ white crystals. Afterward, some of the white crystals were dissolved in 24 mL of benzyl alcohol with constant stirring, and 6 mL of octylamine was added in the above solution and stirred for 30 min. Finally, the resulting solution was transferred to a reaction still heated at 160 °C for 24 h. The obtained precipitates were separated via centrifugation, washed several times with ethanol, and then dried in air at 60 °C for 12 h. The final products $(Y_2O_3:Eu^{3+} nanosheets,$ marked as N1) were prepared by calcination at 800 °C for 2 h. Moreover, Y₂O₃ nanosheets (marked as N2) and Y_2O_3 :Sm³⁺ nanosheets (marked as N3) were obtained by changing the raw material from the above-mentioned procedures [29], whereas YVO₄:Eu³⁺ microsheets (marked as N4) and Y₂O₃:Eu³⁺ microsheets (marked as N5) [30] were obtained by changing both the raw materials and the experimental conditions.

2.2 Preparation of Doped MgB₂-Based Superconductors

The concentration of nano-Ag solution in the experiment was 2000 ppm. The particle size was 15 nm, and the solvent was anhydrous ethanol.

MgB₂ powder and nanosheet/microsheet dopants (MgB₂ powder, microsheet dopant, and nano-Ag solution) were mixed in 15 mL of ethanol to form a suspension. The suspension was transferred into a culture dish after 30 min of sonication. Subsequently, the dish was placed in a vacuum oven for 4 h at 60 °C. The resultant black powder was pressed into tablets. Finally, the tablets were placed in tantalum vessels and annealed at 800 °C for 2 h at heating and cooling rates were of 10 and 5 °C min⁻¹, respectively. Afterward, the final products were obtained. For convenience of description, symbols were used to represent the samples. Furthermore, a pure MgB₂ sample marked as A was prepared for comparison. The symbols, dopant

Table 1Symbols, dopanttypes, and dopant	Symbols of samples	А	В	С	D	Е	F	G	H
concentrations of each sample	Dopant types	None	N2	N3	N1	N4	N5	N5	N5
	Dopant concentrations (wt%)	0	2	2	2	2	2	2	2
	Nano-Ag concentrations (wt%)	0	0	0	0	0	0	0.2	0.4

types, and dopant concentrations of each sample are shown in Table 1.

3 Results and Discussion

Figure 1a–c shows the SEM images of $YVO_4:Eu^{3+}$ microsheets and $Y_2O_3:Eu^{3+}$ microsheets, and the TEM image of $Y_2O_3:Eu^{3+}$ nanosheets, respectively. Figure 1d shows the AFM image of the $Y_2O_3:Eu^{3+}$ nanosheets. Figure 1e presents the thickness of $Y_2O_3:Eu^{3+}$ nanosheets. We know the prepared of $YVO_4:Eu^{3+}$ and $Y_2O_3:Eu^{3+}$

sheets show varying sizes. The sizes of YVO₄:Eu³⁺ and Y₂O₃:Eu³⁺ microsheets are 1–2 and 0.5–1 μ m, respectively, and the size of Y₂O₃:Eu³⁺ nanosheets is approximately 50 nm. The thickness of the Y₂O₃:Eu³⁺ nanosheets is about 2 ~ 3 nm, which is much less than the coherence length of MgB₂.

Figure 2 shows the X-ray diffraction (XRD) patterns of the partial samples and the SEM image of pure MgB₂. XRD results showed that the main phase is MgB₂. A small number of MgO and Mg impurities are also observed, which may broaden the superconducting transition temperature. MgO, which was formed during the preparation of MgB₂,



Fig. 1 SEM images of **a** YVO_4 : Eu³⁺ microsheets (N4), **b** Y_2O_3 : Eu³⁺ microsheets (N5), and **c** TEM image of Y_2O_3 : Eu³⁺ nanosheets (N1); **d** AFM image of the Y_2O_3 : Eu³⁺ nanosheets. **e** Height profile corresponding to the lines draw in **d**

Fig. 2 X-ray diffraction patterns of MgB₂, MgB₂ + 2 wt% Y₂O₃:Eu³⁺ (nm), MgB₂ + 2 wt% Y₂O₃:Eu³⁺ (μ m), and SEM image of pure MgB₂



is present in all samples. We also observed the existence of the Y₂O₃ in the doping samples. The SEM image shows that the irregularly shaped MgB₂ particles are mainly about $0.2-2 \ \mu m$ in size. The boundary between particles is also evident, which also broadens the superconducting transition temperature.

The XRD pattern of MgB₂ doped with $Y_2O_3:Eu^{3+}$ nanosheets is shown in Fig. 2. Because of the low content of $Y_2O_3:Eu^{3+}$ nanosheets, the Y_2O_3 peak is not obvious in the XRD pattern. To further prove that the sample is adulterated with $Y_2O_3:Eu^{3+}$ nanosheets, we performed an elemental analysis of the sample, and results are shown in Fig. 3. Figure 3a presents the SEM image of MgB₂ doped with $Y_2O_3:Eu^{3+}$ nanosheets. Figure 3b–d illustrates the distribution of certain chemical elements; the corresponding element is listed at the top right corner of each figure. According to the element distribution map, the sample contains a large number of Mg, and Y_2O_3 :Eu³⁺ nanosheet inhomogeneous phase dopants distributed around the MgB₂ particles.

Figure 4 depicts the R-T curve of the pure MgB₂ and MgB₂ doped with Y₂O₃:Eu³⁺, Y₂O₃, and Y₂O₃:Sm³⁺ nanosheets. The two characteristic temperatures, namely $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$, on each R-T curve are discussed. (1) The black curve shows the R-T curve of pure MgB₂ (A). The $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$ of pure MgB₂ are 38.2 K and 33.6 K, respectively, the range of superconducting transition temperature is broad, this phenomenon is largely attributed to that the samples contain MgO and Mg impurities and exhibit poor grain connectivity [31]. (2) The resistivity of MgB₂ doped with

Fig. 3 SEM image of a MgB₂ + 2 wt% Y₂O₃:Eu³⁺ (nm) (D) and chemical element distribution map (**b–d**)





Fig. 4 Temperature-dependent resistivity of MgB_2 doped with different nano-sheets (pure MgB_2 (A), $MgB_2 + 2$ wt% Y_2O_3 (nm) (B), $MgB_2 + 2$ wt% Y_2O_3 :Sm³⁺ (nm) (C), $MgB_2 + 2$ wt% Y_2O_3 :Eu³⁺ (nm) (D))

nanosheets is higher than that of pure MgB₂. (3) The $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃:Eu³⁺ nanosheets increases by 1.2 K compared with that of pure MgB₂, which may be due to the Y₂O₃:Eu³⁺ nanosheets EL material distributed around the MgB₂ particles to form a special response meta-structure. Y₂O₃:Eu³⁺ nanosheets would generate an EL during the measurement of R-T curve of the sample, which may improve the superconducting transition temperature [19]. (4) The $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃:Sm³⁺ nanosheets are lower than that of the pure MgB₂ sample. The MgB₂ doped with Y₂O₃:Sm³⁺ nanosheets is lower than that of the pure MgB₂. The $T_{\rm C}^{\rm off}$ values of MgB₂ doped with Y₂O₃ and Y₂O₃:Eu³⁺ nanosheets are reduced by 0.2 K, and MgB₂ doped with Y₂O₃:Sm³⁺ nanosheets is reduced by 0.4 K.

Smolyaninov et al. proposed that MgB2 doped with 5-nm diamond particles can make the dielectric constant close to 0; consequently, the superconducting transition temperature of the MgB₂-based metamaterial superconductor can reach the liquid nitrogen temperature [32]. On the basis of the idea that changing the effective dielectric constant can increase the superconducting transition temperature of a metasuperconductor, we prepared MgB₂ doped with Y₂O₃:Eu³⁺ microsheets and nano-Ag solution to change the dielectric constant of the system so as to improve the superconducting transition temperature of MgB₂. Figure 5 presents the R-T curve of the MgB₂ doped with Y_2O_3 :Eu³⁺ microsheets and nano-Ag solution. However, this graph indicated that the MgB₂ doped with Y₂O₃:Eu³⁺ microsheets and nano-Ag solution fails to improve the $T_{\rm C}$ of MgB₂. All of the doped samples exhibit superconducting transition. The resistivity in the normal state increases, and the superconducting



Fig. 5 Temperature-dependent resistivity of MgB₂ doped with $Y_2O_3:Eu^{3+}$ microsheets and nano-Ag solution (MgB₂ (A), MgB₂ + 2 wt% $Y_2O_3:Eu^{3+}$ (μ m) + 0.2 wt% Ag (G), MgB₂+2 wt% $Y_2O_3:Eu^{3+}$ (μ m) + 0.4 wt% Ag (H)

transition temperature of MgB₂ doped with nano-Ag solution decreases remarkably.

Figure 6 presents the R-T curves of six different doped samples under different currents, the upper left and bottom right corners are enlarged graphs of corresponding $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$, respectively. Table 2 provides a list of the concrete values of $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$. Figure 7 shows the relationship between the $T_{\rm C}^{\rm off}$ of doped samples and currents. According to the results presented in Figs. 6, 7, and Table 2, the following trends are determined: (1) With increasing current, the $T_{\rm C}^{\rm off}$ values of pure MgB₂ (A) and MgB₂ doped with Y₂O₃ nanosheets (B) and Y₂O₃:Sm³⁺ nanosheets (C) decrease, and the $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y_2O_3 nanosheets and Y_2O_3 :Sm³⁺ nanosheets are lower than those of pure MgB₂ at the corresponding current; this result is attributed to impurity doping. (2) The T_C^{off} of MgB₂ doped with Y₂O₃:Eu³⁺ micro-sheets (F), Y₂O₃:Eu³⁺ nanosheets (D), and YVO₄:Eu³⁺ micro-sheets (E) show a slight increase at a current less than or equal to 100 mA. However, when the current is larger than 100 mA, T_{C}^{off} decreases with the increasing current. This result may be due to the considerably high current, which results in a remarkable thermal effect. Consequently, the temperature gradient in the superconducting sample is increased, thereby increasing the influence of thermoelectric potential on the measurements [25]. (3) The $T_{\rm C}^{\rm on}$ of the pure MgB₂ sample remains unchanged when increasing current, whereas that of the doped samples show a slight increase. The T_C^{on} values of all nonluminous inhomogeneous phase doping samples are no more than 38.2 K, but those of samples doped with EL inhomogeneous phases are more than 38.2 K at a certain current. Japanese scientists observed superconducting transition by changing the electric field in



Fig. 6 Temperature-dependent resistivity of doped samples under different currents (50, 100, 200, 300, 500 mA)

off on on							
Table 2 The $T_{\rm C}^{\rm off}$ and $T_{\rm C}^{\rm off}$ of doped samples under different		A/K	B/K	C/K	D/K	E/K	F/K
currents	50 mA	33.8-38.2	33.6-37.6	33.4-37.6	34.6-37.8	34.6-37.8	34.2–38
	100 mA	33.6-38.2	33.4–38	33.2-37.8	34.8-38	35.2-38	34.6-38
	200 mA	33.4-38.2	33–38	33–38	34.4-38.2	34.8-38	34.4–38
	300 mA	33.2-38.2	32.8-38	32.8-38	34.2-38.4	34.6-38	34.2-38.2
	500 mA	33-38.2	32.6–38	32.6–38	34-38.4	34.2-38.2	34-38.4



Fig. 7 The relationship between the T_C^{off} of doped samples and currents

unconventional superconductors ZrNCl and MoS₂. ZrNCl and MoS₂ show $T_{\rm C}$ of 15.2 and 10.8 K, respectively, on the optimum carrier doping [27, 28]. Nevertheless, in our experiment, the effect of changing electric field on the $T_{\rm C}$ is not obvious; this may be because we change the carrier density by changing the current directly rather than changing the electric field to induce carrier density change.

4 Conclusion

On the basis of the smart metamaterial superconductor model, we found that the inhomogeneous phase significantly improves the $T_{\rm C}$ of a superconductor. In this paper, the responses of the $T_{\rm C}$ of MgB₂ to inhomogeneous phase doping and changing the applied electric field are systematically investigated. At first, we prepared Y_2O_3 :Eu³⁺ and Y_2O_3 , Y_2O_3 :Sm³⁺ nanosheet inhomogeneous phases, which were doped into MgB₂ by an ex situ process. Results showed that the $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃:Eu³⁺ nanosheets increase by 1.2 K compared with that of pure MgB₂, whereas the $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃ and Y_2O_3 :Sm³⁺ nanosheets decrease, and the MgB₂ doped with $Y_2O_3:Sm^{3+}$ nanosheets presents the lowest T_C^{off} . In addition, the $T_{\rm C}^{\rm on}$ of all of the MgB₂ doped with nanosheets decrease. The distribution of certain chemical elements reveals that Y₂O₃:Eu³⁺ nanosheets inhomogeneous phase dopants distributed around the MgB₂ particles and formed a meta-structure. Hence, the effectiveness of Y_2O_3 :Eu³⁺ nanosheets which improves the T_C of MgB₂ can be fully reflected. Then, on the basis of the idea that changing the effective dielectric constant can increase the superconducting transition temperature of a meta-superconductor, we prepared MgB₂ doped with Y₂O₃:Eu³⁺ microsheets and nano-Ag solution to change the dielectric constant of the system so as to improve the superconducting transition temperature of MgB₂. Nevertheless, experimental results show that the codoping cannot improve the $T_{\rm C}$ of MgB₂. Additionally, the superconducting transition temperature of MgB₂ doped with nano-Ag solution decreases remarkably, and the resistivity in the normal state increases. Finally, we also find that the applied electric field affects the $T_{\rm C}$ of doping samples; when increasing the test current, the T_C^{off} of nonluminous inhomogeneous phase doping samples decrease. However, the T_C^{off} of luminescent inhomogeneous phase doping samples increase and then decrease. The T_C^{on} of pure MgB₂ showed no change, whereas the $T_{\rm C}^{\rm on}$ of doped samples can more than 38.2 K at certain conditions. Improving the superconducting transition temperature of MgB₂ can not only increase its application but also promote the development of superconductivity theory. This study provides a further exploration for the considerable challenge of improving the $T_{\rm C}$ of smart meta-superconductor MgB₂.

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