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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 MgB₂ · Y₂O₃:Eu³⁺ nanosheets · Inhomogeneous phase · Applied electric field·*T*_C

1 Introduction

Since Akimitsu et al. [\[1\]](#page-6-0) 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 $(T_{\rm C} = 39 \text{ K})$ [\[2\]](#page-6-1). 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 $T_{\rm C}$ of MgB₂ [\[3](#page-6-2)[–7\]](#page-6-3). In addition, another possible method for improving the $T_{\rm C}$ 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 $[8, 9]$ $[8, 9]$ $[8, 9]$. 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 $T_{\rm C}$ 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 [\[10–](#page-6-6)[15\]](#page-6-7). With the development of meta-material, the use of meta-material concept to design superconducting materials to improve its T_C has been recognized. We proposed the introduction of electroluminescence (EL) materials in meta-materials to enhance the superconducting transition temperature through EL [\[16,](#page-7-0) [17\]](#page-7-1). Zhang et al. [\[18\]](#page-7-2) 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 Y_2O_3 :Eu³⁺ EL material to form the impurity phase of YB4. To avoid the formation of the impurity phase of YB₄, Tao et al. [\[19\]](#page-7-3) doped Y₂O₃:Eu³⁺

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EL material into MgB_2 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 Y₂O₃: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 $\text{YVO}_4:\text{Eu}^{3+}$ microsheets EL material on the superconducting transition temperature of MgB_2 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. $[20-24]$ $[20-24]$ proposed that material can be designed as a meta-material structure with an effective dielectric constant $\varepsilon_{\text{eff}} \approx 0$, which can improve the T_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 $[25, 26]$ $[25, 26]$ $[25, 26]$. Ye et al. $[27, 28]$ $[27, 28]$ $[27, 28]$ 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_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 MgB_2 particles are used as the matrix material; the EL material distributed around the MgB_2 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_c [\[18,](#page-7-2) [19\]](#page-7-3). 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 Y_2O_3 : Eu³⁺, Y_2O_3 , and Y_2O_3 : Sm³⁺ nanosheet inhomogeneous phases; they were doped into $MgB₂$ by an ex situ process. The effects of doping $Y_2O_3:Sm^{3+}/Y_2O_3$ nonluminous inhomogeneous phase or $Y_2O_3:Eu^{3+}$ 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 Y_2O_3 and Eu_2O_3 powder were added to 4 mL of concentrated nitric acid under stirring and heating at 70 °C for 1 h to obtain the $Y(Eu)(NO_3)$ 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, Y_2O_3 nanosheets (marked as N2) and $Y_2O_3:Sm^{3+}$ nanosheets (marked as N3) were obtained by changing the raw material from the above-mentioned procedures [\[29\]](#page-7-10), whereas YVO_4 :Eu³⁺ microsheets (marked as N4) and Y_2O_3 :Eu³⁺ microsheets (marked as N5) [\[30\]](#page-7-11) 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 \degree 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

types, and dopant concentrations of each sample are shown in Table [1.](#page-2-0)

3 Results and Discussion

Figure [1a](#page-2-1)–c shows the SEM images of $\text{YVO}_4: \text{Eu}^{3+}$ microsheets and Y_2O_3 :Eu³⁺ microsheets, and the TEM image of Y_2O_3 :Eu³⁺ nanosheets, respectively. Figure [1d](#page-2-1) shows the AFM image of the Y_2O_3 :Eu³⁺ nanosheets. Figure [1e](#page-2-1) presents the thickness of Y_2O_3 : Eu³⁺ nanosheets. We know the prepared of YVO₄: Eu^{3+} and Y₂O₃: Eu^{3+}

sheets show varying sizes. The sizes of $YVO_4:Eu^{3+}$ and Y₂O₃:Eu³⁺ microsheets are 1–2 and 0.5–1 μ m, respectively, and the size of Y_2O_3 : Eu^{3+} nanosheets is approximately 50 nm. The thickness of the Y_2O_3 :Eu³⁺ nanosheets is about 2 \sim 3 nm, which is much less than the coherence length of MgB2.

Figure [2](#page-3-0) 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** YVO₄:Eu³⁺ microsheets (N4), **b** Y₂O₃:Eu³⁺ microsheets (N5), and **c** TEM image of Y₂O₃: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 MgB_2 , $MgB_2 + 2$ wt% Y_2O_3 :Eu³⁺ (nm), $MgB_2 + 2$ wt% Y_2O_3 :Eu³⁺ (*μ*m), and SEM image of pure $MgB₂$

is present in all samples. We also observed the existence of the Y_2O_3 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 Y₂O₃:Eu³⁺ nanosheets is shown in Fig. [2.](#page-3-0) Because of the low content of Y₂O₃:Eu³⁺ nanosheets, the Y₂O₃ peak is not obvious in the XRD pattern. To further prove that the sample is adulterated with Y_2O_3 :Eu³⁺ nanosheets, we performed an elemental analysis of the sample, and results are shown in Fig. [3.](#page-3-1) Figure [3a](#page-3-1) presents the SEM image of $MgB₂$ doped with Y_2O_3 :Eu³⁺ nanosheets. Figure [3b](#page-3-1)-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 MgB_2 particles.

Figure [4](#page-4-0) depicts the $R - T$ curve of the pure MgB₂ and MgB₂ doped with Y_2O_3 :Eu³⁺, Y_2O_3 , and Y_2O_3 :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_C^{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** $MgB_2 + 2 wt\% Y_2O_3:Eu^{3+}$ (nm) (D) and chemical element distribution map (**b**–**d**)

Fig. 4 Temperature-dependent resistivity of MgB₂ doped with different nano-sheets (pure $MgB_2(A)$, MgB_2+2 wt% Y_2O_3 (nm) (B), $MgB_2 + 2$ wt% $Y_2O_3:Sm^{3+}$ (nm) (C), $MgB_2 + 2$ wt% $Y_2O_3:Eu^{3+}$ $(nm)(D))$

nanosheets is higher than that of pure MgB₂. (3) The $T_{\rm C}^{\rm off}$ of MgB_2 doped with Y₂O₃:Eu³⁺ nanosheets increases by 1.2 K compared with that of pure MgB2, which may be due to the Y_2O_3 :Eu³⁺ nanosheets EL material distributed around the $MgB₂$ particles to form a special response meta-structure. Y_2O_3 :Eu³⁺ nanosheets would generate an EL during the measurement of *R*–*T* curve of the sample, which may improve the superconducting transition temperature [\[19\]](#page-7-3). (4) The $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃ and Y₂O₃:Sm³⁺ nanosheets are lower than that of the pure $MgB₂$ sample. The MgB₂ doped with $Y_2O_3:Sm^{3+}$ nanosheets presents the lowest $T_{\rm C}^{\rm off}$. The $T_{\rm C}^{\rm on}$ of all of the MgB₂ doped with nanosheets is lower than that of the pure MgB2. The $T_{\rm C}^{\rm on}$ values of MgB₂ doped with Y₂O₃ and Y₂O₃:Eu³⁺ nanosheets are reduced by 0.2 K, and MgB₂ doped with Y_2O_3 :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 Y_2O_3 :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](#page-4-1) 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_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 :Eu³⁺ microsheets and nano-Ag solution (MgB₂ (A), MgB₂ + 2 wt% Y₂O₃:Eu³⁺ (μ m) + 0.2 wt% Ag (G), MgB₂ + 2 wt% Y₂O₃:Eu³⁺ $(\mu m) + 0.4$ wt% Ag (H)

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

Figure [6](#page-5-0) 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](#page-5-1) provides a list of the concrete values of $T_{\rm C}^{\rm on}$ and $T_{\rm C}^{\rm off}$. Figure [7](#page-6-8) shows the relationship between the T_C^{off} of doped samples and currents. According to the results presented in Figs. [6,](#page-5-0) [7,](#page-6-8) and Table [2,](#page-5-1) 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 Y₂O₃ nanosheets and Y₂O₃:Sm³⁺ nanosheets are lower than those of pure $MgB₂$ at the corresponding current; this result is attributed to impurity doping. (2) The $T_{\rm C}^{\rm off}$ of MgB₂ doped with Y₂O₃:Eu³⁺ micro-sheets (F), Y_2O_3 :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, $T_{\rm C}^{\rm off}$ 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 [\[25\]](#page-7-6). (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 $T_{\rm C}^{\rm on}$ 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)

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

unconventional superconductors $ZrNCl$ and $MoS₂$. $ZrNCl$ and $MoS₂$ show T_C of 15.2 and 10.8 K, respectively, on the optimum carrier doping [\[27,](#page-7-8) [28\]](#page-7-9). 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 MgB_2 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_C^{off} of MgB₂ doped with Y₂O₃ and Y_2O_3 :Sm³⁺ nanosheets decrease, and the MgB₂ doped with Y_2O_3 :Sm³⁺ 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 Y_2O_3 :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 Y_2O_3 :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_{C} of doping samples; when increasing the test current, the $T_{\rm C}^{\rm off}$ of nonluminous inhomogeneous phase doping samples decrease. However, the $T_{\rm C}^{\rm off}$ of luminescent inhomogeneous phase doping samples increase and then decrease. The $T_{\rm C}^{\rm on}$ of pure MgB_2 showed no change, whereas the T_C^{on} of doped samples can more than 38.2 K at certain conditions. Improving the superconducting transition temperature of MgB_2 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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