

# Superconductivity of $\text{MgB}_2$ Layers Prepared on Silicon Substrate by Implantation of Magnesium Ions into Boron Substrate

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The results of investigation of the  $\text{MgB}_2$  layers prepared on silicon substrate by implantation of Mg ions into boron substrate are presented. After implantation the annealing processes were carried out at temperatures 673 K, 773 K, and 873 K in a furnace in an atmosphere of flowing Ar-4% $\text{H}_2$  gas mixture. The samples were characterized by: four-probe electric conductivity measurements and magnetically modulated microwave absorption. Our results showed that due to silicon substrate the diffusion of implanted Mg ions into boron materials should be limited, and the superconducting phase forms a continuous  $\text{MgB}_2$  layer and the resistivity for all samples fall down to zero below  $T_c$ . The transition temperature  $T_c$  becomes higher with increasing annealing temperature:  $T_c = 18$  K (for annealing at  $T_A = 673$  K),  $T_c = 20$  K (for annealing at  $T_A = 773$  K), and  $T_c = 27$  K (for annealing at  $T_A = 873$  K).

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## 1. Introduction

Magnesium borides have been studied from 19th century [1], but the discovery in 2001 of the superconductivity of  $\text{MgB}_2$  [2], having a remarkably high critical temperature  $T_c = 39$  K, stimulated a world wide research of the properties of  $\text{MgB}_2$  because of the potential of technical applications of these new superconducting materials. Much effort has been taken to obtain thin film of  $\text{MgB}_2$  with a good quality and high  $T_c$  and high critical current [3–10]. Our previous papers show results of the investigation of evolution of the superconducting regions in  $\text{MgB}_2$  which forms separate islands of superconducting phase [6, 10–14]. We applied a few techniques: four-probe electric conductivity measurements, magnetically modulated microwave absorption (MMA), magnetometric measurements, and the Rutherford backscattering spectroscopy (RBS). It is of fundamental importance to establish whether there is a limit to how dimension of superconducting island or how thin a superconducting wire can be, while retaining its superconducting character [15, 16]. To synthesize  $\text{MgB}_2$  superconducting phase we used the ion implantation technique: boron implanted magnesium or magnesium implanted boron substrates, followed by transient annealing using a high intensity pulsed plasma

beam or by conventional thermal annealing in the temperature range from 673 K to 873 K in a stream of flowing Ar-4% $\text{H}_2$  mixture. In all samples the superconducting phase does not form a continuous layer and a resistivity does not fall down to zero. Apparently separate islands of superconducting phase were formed and samples were below the percolation threshold.

To continue previous studies, in this paper we show how to improve the technology of ions implantation to obtain homogeneous thin layers of  $\text{MgB}_2$  in the percolation limit.

## 2. Experimental

Samples of superconducting  $\text{MgB}_2$  layers were obtained by the following process. At first boron films of the thickness 800 nm were deposited onto silicon substrates. Then, those substrates were implanted with Mg in triple mode, i.e. each sample was sequentially implanted at three different energies starting from 140 keV, through 80 keV to 40 keV. To get stoichiometry appropriate for  $\text{MgB}_2$ , computer simulations using the SUSPRE program were performed for multi-energy implantations. The ion fluence was:  $6 \times 10^{17}$  ions/ $\text{cm}^2$  at 140 keV,  $3 \times 10^{17}$  ions/ $\text{cm}^2$  at 80 keV and 40 keV. The maximum thickness of Mg layer was 300 nm. Then, the samples were heated for 20 min at different temperatures, i.e. at 673 K, 773 K, and 873 K, to remove radiation damage and stabilize the reaction in the solid state. To apply

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implantation of Mg ions into boron films of the thickness 800 nm deposited onto silicon substrates, instead of bulk boron substrate, the diffusion of implanted Mg ions is limited.

We have studied the quality of superconducting properties of samples using four-probe electric conductivity measurements and MMMA. The measurements of electric conductivity were performed in a helium flow cryostat enabling the temperature to be decreased from 300 K down to 4 K. The temperature was controlled with  $\pm 0.1$  K accuracy by an Oxford Instruments ITC-503 unit. MMMA measurements were performed using standard X-band (9.4 GHz) EPR spectrometer with a helium flow cryostat.

### 3. Results

#### 3.1. Resistivity measurements

The temperature dependence of resistivity for the three samples annealed at different temperatures, i.e. at 673 K, 773 K, and 873 K is shown in Fig. 1. Below  $T_c$  the resistivity in all samples drop to zero value, which shows the percolation limit of superconducting islands. We have obtained a good quality of thin layers of  $\text{MgB}_2$ . For annealing temperatures 673 K, 773 K, and 873 K the transition temperatures ( $T_c$  onset) are: 18 K, 20 K, and 27 K, respectively. The width of the transition temperature  $\Delta T_c$  is 2 K for samples annealed at 673 K and 773 K. Only the sample with the highest  $T_c(\text{onset}) = 27$  K has  $\Delta T_c = 5$  K.

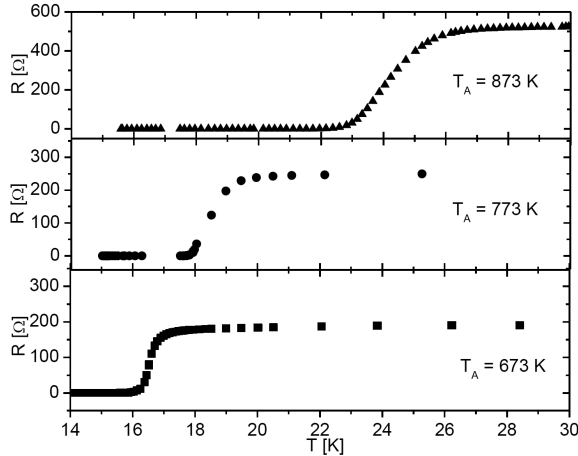


Fig. 1. The temperature dependence of the resistance for three different annealed temperatures.

#### 3.2. Magnetically modulated microwave absorption

The MMMA technique, introduced by Stankowski [17], is a perfect method to determine temperature of the transition to the superconducting state  $T_c$ , especially for samples with nanometric scale islands of the superconducting phase, below the percolation limit. Figure 2 presents the

temperature dependence of MMMA for three samples annealed at different temperatures. We have obtained, in similar to resistivity measurements, the transition temperature  $T_c$  onset: 18 K, 20 K, and 27 K for the samples annealing at temperatures 673 K, 773 K, and 873 K, respectively. It is clearly seen that there is a strong correlation between the temperature of annealing samples  $T_A$  and  $T_c$ . Therefore, the highest annealing temperature resulted in the highest  $T_c$ .

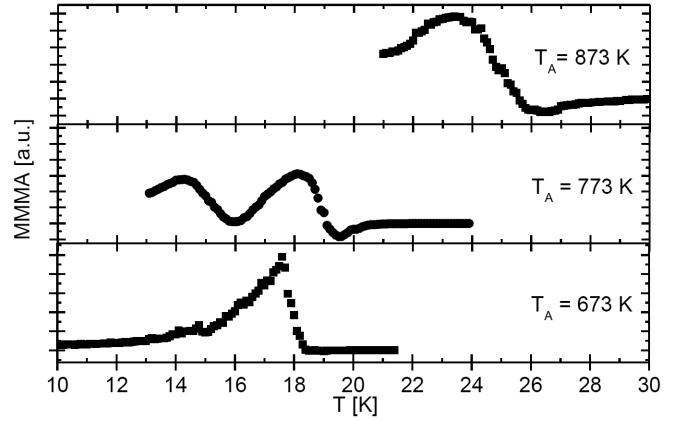


Fig. 2. The temperature dependence of MMMA for three different annealed temperatures.

### 4. Discussion and conclusions

The technique of Mg ion implantation into boron films of the thickness 800 nm onto silicon substrates, instead of bulk boron substrate, allows us to get good quality of the  $\text{MgB}_2$  thin layers. The diffusion of implanted Mg ions was limited, and we have obtained thin layers of  $\text{MgB}_2$  above the percolation limit. Below  $T_c$  the resistivity was equal to zero. Why we do not get the transition temperature  $T_c = 39$  K characteristic for the bulk  $\text{MgB}_2$  superconductor? The transition temperature of the superconducting state  $T_c$  decreases greatly, as the thickness of  $\text{MgB}_2$  film decreases from 35 K for 200 nm thick film to 10 K for 5 nm thick film [9]. The thin film of  $\text{MgB}_2$  is not homogeneous but composed of grains (islands) and the Josephson junctions are realized. Finally, we are able to detect MMMA. When the average distance between the islands decreases, the Josephson coupling increases and superconducting percolation is achieved. For the sample annealing at 773 K, MMMA shows two thicknesses of films with  $T_c$  equal to 20 K and 16 K, or separately small isolated islands of  $T_c = 16$  K. Resistivity measurements show only one  $T_c = 20$  K, because the percolation is achieved and resistance of sample is equal to zero.

The results of investigations of the  $\text{MgB}_2$  thin films samples, described in this paper, obtained by Mg ions implantation into boron films of the thickness 800 nm onto silicon substrates followed by temperature treatment, can be summarized as follows:

1. Increase of the temperature of annealing  $\text{MgB}_2$  thin films gives higher  $T_c$ .
2. To apply implantation of Mg ions into boron films of the thickness 800 nm deposited onto silicon substrates, instead of bulk boron substrate, the diffusion of implanted Mg ions is limited, and percolation is achieved. The resistance of  $\text{MgB}_2$  thin films samples below  $T_c$  is equal to zero.

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