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Formation of Superconducting Regions of MgB_2 by Implantation of Boron Ions into Magnesium Substrate

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The results of investigation of the MgB_2 inter-metallic compound with the use of boron ions implantation and plasma pulse treatment are presented. The samples were characterized by: four-probe electric conductivity measurements, magnetically modulated microwave absorption, and magnetic measurements. For hydrogen and argon pulsed plasma treatment the samples with T_c ranging from 10 K to 32 K were obtained. The superconducting phase does not form a continuous layer since the resistivity does not fall down to zero. Apparently, separate islands of superconducting phase are connected through metallic Mg paths. All samples are still below the percolation threshold.

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1. Introduction

The discovery of superconductivity in MgB_2 with T_c as high as 39 K [1] has created great excitement about the possibility of using this material in many practical applications. Soon after the discovery, the first superconducting MgB_2 thin films were successfully demonstrated in the literature [2–4]. The goal of the studies undertaken by present authors is to synthesize for the first time, superconducting MgB_2 film from liquid phase without annealing in Mg vapor, using ion implantation and transient melting processes (TMP). For TMP one can use short laser, electron, ion, and plasma pulses. In the present work boron ions of energy 100 keV and doses of 5×10^{18} ions B/cm^2 were implanted into magnesium substrates. For TMP the hydrogen and argon plasma pulses of energy densities between 1.8–3.2 J/cm^2 and 2–4 number of pulses were used.

2. Experimental

Three experimental methods were used to study superconducting behavior of thin layers of MgB_2 formed from the solid phase in the Mg–B system: magnetic measurements (Oxford Instruments MagLab 2000 System DC magnetometer/AC susceptometer), magnetically modulated microwave absorption (MMMA), and four-probe electric conductivity measurements. The synthesis of the superconducting MgB_2 samples were made by means of a transient melting process. This method omits necessity of annealing the samples in the Mg vapor.

The samples before TMP treatment were characterized by access on the surface of the samples. These samples were not superconducting before TMP process. To obtain MgB_2 superconducting layer, the TMP process was conducted with H_2 and Ar plasma pulses.

In order to get inside into heat evolution within the substrate subjected to the pulse plasma treatment we calculated temperature versus time and melt depth in pure Mg irradiated with plasma pulses of a few J/cm^2 energy density. Samples were plasma irradiated in the following way: sample #33 — two H_2 plasma pulses of 1.8 and 1.9 J/cm^2 energy, sample #49 — two H_2 plasma pulses of 3.2 and 2.9 J/cm^2 energy, and sample #59 — four Ar plasma pulses of 1.8 J/cm^2 energy.

3. Results

The results are presented in such a way as to show the impact of TMP process on the formation of superconducting phase in a layer. The confirmation that the zero field MMMA line is related to the superconducting state implies observation of the Josephson hysteresis loop (JHL). The JHL has an opposite circulation versus magnetic field B to the typical magnetic hysteresis loop. Figure 1a shows MMMA signals for sample #49 as a function of a magnetic field. At the temperature $T = 30$ K there is no hysteresis loop for sample #49. This

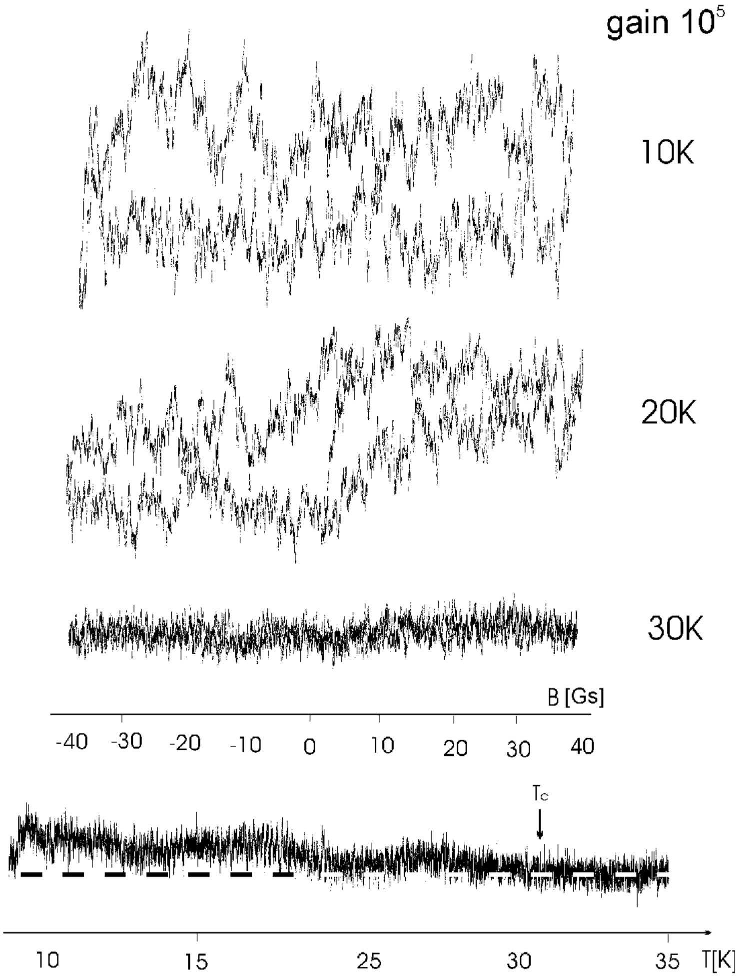

Fig. 1. Magnetically modulated microwave absorption signal vs. (a) external magnetic field, (b) temperature for the sample #49 of Mg by implantation of boron ions treated with two hydrogen plasma pulses of 3.2 J/cm^2 and of 2.9 J/cm^2 (thin film).

means that superconducting phase vanishes near this temperature. Figure 1b confirms that the temperature of superconducting-normal state is apart 32 K. Stronger signals from superconducting phase were observed for sample #59. The intensity of the hysteresis loop in that sample is 10 times greater than for sample #49. The critical temperature, however, is only 12 K (Fig. 2).

Magnetic moment versus temperature $m(T)$ when zero field cooled for the sample #59 is illustrated in Fig. 2b. Electrical conductivity shows a distinct difference between plasma pulses of H_2 and Ar. Figure 3 presents the percentage of the electric resistance in the dependence on temperature. This figure shows the

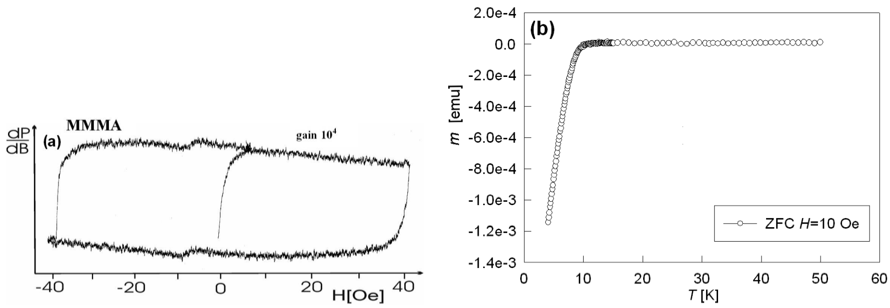

Fig. 2. Josephson hysteresis loop at $T = 10$ K (a) and magnetic moment versus temperature $m(T)$ (b) for the sample #59 of Mg by implantation of boron ions treated with four argon plasma pulses of 1.8 J/cm^2.

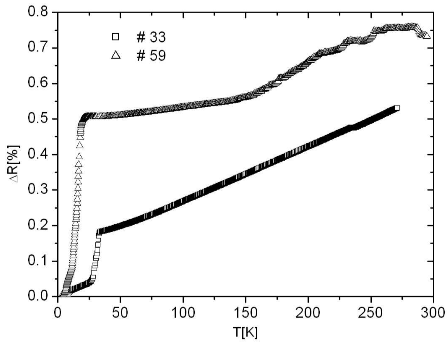

Fig. 3. The temperature dependences of the resistance for the samples of Mg by implantation of boron ions treated with: □ two hydrogen plasma pulses of 1.8 J/cm^2, sample #33, Δ four argon plasma pulses of 1.8 J/cm^2, sample #59.

difference of samples #33 (TMP hydrogen) and #59 (TMP argon). Figure 3 also shows the difference in the critical temperature T_c between samples #33 and #59.

4. Discussion

Figures 1, 2, 3 were drawn for different processes of TMP. The use of high enough melting of Mg layer implants with the excess of boron leads to the small island-like areas of superconducting phase MgB_2. This phase is formed as results of boron ions diffusion into Mg layer to form MgB_2. The areas of MgB_2 are formed and T_c close to the T_c of bulk is obtained (39 K). Figures 1 and 2 show that T_c for samples #33 and #44 are close to 32 K. Low energy melting process of magnesium with several impulses (sample #59, Fig. 2) does not allow boron atoms to diffuse into the whole Mg layer. This results in formation of large superconducting island (strong MMMA signals) of nonstoichiometric Mg_xB_{1-x}. For films with higher boron concentration T_c is much lower than in bulk sample. Critical temperatures are around 10 K [5]. Sample #59 is characterized by a strong MMMA signal with low T_c (Fig. 2a and b).

Electrical conductivity studied in sample #33 showed anisotropic behavior. The resistivity measurements at perpendicular orientation of the implanted layer exhibits semiconducting properties, whereas at parallel orientation metallic characteristic is observed between islands of Mg_xB_{1-x}. The resistivity does not drop to zero value, which shows that this is no percolation for superconducting islands in metallic magnesium layer.

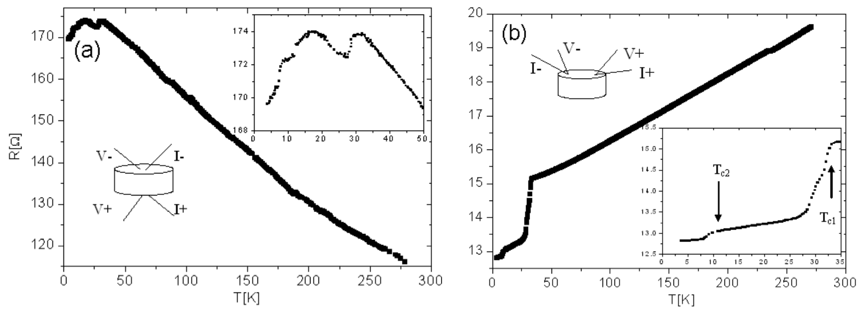

Fig. 4. The temperature dependences of the resistance for the sample #33 of Mg by implantation of boron ions treated with two hydrogen plasma pulses of 1.8 J/cm^2 measured in direction perpendicular to (a) and in the plane (b) of the sample. Insets: magnifications of the resistance curves below T_c.

Anisotropy of conductivity is presented in Fig. 4. Figure 4b for sample #33 shows two drops in conductivity at $T_{c1} \approx 32$ K and $T_{c2} \approx 10$ K. This is a result of coexistence of two superconducting phases generated in TMP process.

5. Conclusions

Mg layers implanted with boron ions in TMP process exhibit critical temperature much lower than bulk MgB_2 material, even as low as 10 K, if this is an excess of boron relative to magnesium [5]. Higher energies of hydrogen plasma applied up to 3 J/cm^2 in TMP cause the melting process in thicker layer of Mg implanted with B. The critical temperature is then higher than in low energy plasma pulses. However, in these samples the superconducting islands are much smaller (weak MMMA signals) and no percolation results in a drop of conductivity to non-zero value. The application of low energies in melting process (argon plasma pulses) gives better properties of superconducting material. The adjustment of TMP parameters is very crucial in generating more spacious regions of superconducting phase in near surface of the layer. The optimization of these parameters in TMP process to get a superconducting film is still in a progress.

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