Magneto-Optical Studies of the Critical States in c-axis Oriented MgB_2 Thin Film and Bulk MgB_2/Mg Nano-Composites

Zuxin Ye, Qiang Li, G. D. Gu, J. J. Tu, W. N. Kang, Eun-Mi Choi, Hyeong-Jin Kim, and Sung-Ik Lee

***Abstract*—We present magneto optical (MO) studies of the critical states in a c-axis oriented MgB_2 thin film with a critical current density (J_c) of 6 MA/cm^2 at 5 K, and bulk MgB_2/Mg nano-composites with J_c over 1 MA/cm^2 at 5 K. The magnetic flux penetration and trapping in the thin film are found remarkably different from that in the bulk samples. A tree-like magnetic flux pattern was observed entering the superconducting film, as well as various complex dendritic flux jumps due to the thermo-magnetic instability. These behaviors indicate the collapse of the critical state of a type II superconductor at local level. In contrast, a regular flux penetration into the critical state was observed in the bulk nano-composites, which indicates that the addition of pure Mg helps to stabilize the critical state.**

***Index Terms*—Critical state, flux jump, magneto-optical, superconductors.**

I. Introduction

THE recently discovered superconducting MgB_2 [1] has a great potential for practical applications. Soon after the discovery, both MgB_2 thin film [2], [3] and wires [4] with high critical current density (J_c) were fabricated with rather conventional processing methods. On the other hand, flux jumps associated with the kinks (or noise) on the magnetic hysteresis loop were also observed in the high J_c MgB_2 samples at low temperatures. The behavior of flux jump is related to the thermo-magnetic instability of the motion of magnetic vortices in a type II superconductor. It is well known that flux jump can result in a large-scale flux avalanche in the critical state [5], which could have a devastating consequence in such practical applications as energy storage, power transmission. In this work, we present magneto-optical (MO) studies of the critical states in a c-axis oriented MgB_2 thin film with J_c of 6 MA/cm^2 at 5 K and self-field, and the bulk MgB_2/Mg nano-composites with J_c over 1 MA/cm^2 at 5 K and self-field. The magnetic flux penetration and trapping in the thin film are found remarkably different from that in the bulk samples. A tree-like magnetic flux pattern and various complex dendritic flux jumps were observed entering and exiting the superconducting film. In contrast, a regular flux penetration was observed in the bulk nano-composites, which indicates that the addition of pure Mg likely helps to stabilize flux motion.

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II. Experimental Details

A. c-Axis Oriented MgB_2 Thin Film

For this study, high quality c-axis oriented MgB_2 thin films (~450 nm thick) were deposited on c-cut Al_2O_3 substrate using a pulsed laser deposition method as described previously [2]. The superconducting transition $(T_c = 39.6\ \mathrm{K})$ was found extremely sharp with $\delta T_c < 0.1$ K by a standard four-probe resistive technique. J_c at zero field was $\sim 6 \times 10^6$ A/cm^2 at 5 K and $\sim 3 \times 10^5$ A/cm^2 at 35 K by applying the Beam model to the magnetization hysteresis M–H loop.

B. MgB_2/Mg Superconducting Nano-Composites

MgB_2/Mg composites were prepared using 99.99% Mg ingot and 99.99% B powder in an alumina crucible heated in a temperature gradient 600–1200°C. Standard four-probe resistivity measurements showed that the pellets have a sharp transition at 39 K. Detailed Transmission Electron Microscopy (TEM) characterization showed that the polycrystalline sample was extremely dense and had a grain size on the order of tens of nm. Both bulk magnetization and transport measurements showed that J_c at zero field was over 10^6 A/cm^2 at 5 K and $\sim 3 \times 10^5$ A/cm^2 at 30 K. Considering the fact that grains in these composites are randomly oriented, as compared to the c-axis thin film used for this study, the composites are capable of carrying very high critical currents.

C. MO Experimental Setup

The MO experimental set up is similar to that used by Polyanski *et al.* [5]. The garnet MO indicator film with in-plane magnetization was placed directly onto the sample surface. Magnetic field was applied perpendicular to the sample surface. For nano-composites, the grain size is below the resolution limit of MO technique $(\sim 2\ \mu\mathrm{m})$. Flux penetration to individual grains can not be directly imaged. However, electromagnetic granularity of a bulk specimen can be determined from the global MO images taken on the magnetic shielding state to conclude whether the grain boundaries are strongly or weakly coupled at the applied external field.

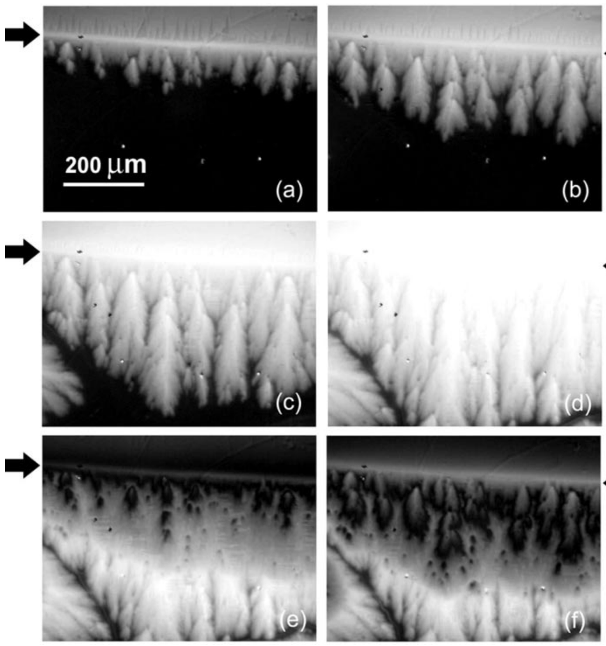

Fig. 1. MO images of flux penetration and trapping into zero-field-cooled MgB_2 thin film at 5.5 K, where arrows indicating the film edge. The respective images were taken at $H = 100$, 250, 450, 650, 300, and 0 Oe during a field increasing and decreasing cycle. At ~600 Oe, full penetration was reached (d).

III. Results and Discussions

A. Flux Penetration and Trapping in the c-axis Oriented MgB_2 Thin Film

Fig. 1 is a sequence of MO images taken during a field cycle at 5.5 K, showing flux penetration and trapping into zero-field-cooled MgB_2 thin film. The image brightness represents flux density. The arrows in the figure indicate the film edge. The respective images were taken at $H = 100$, 250, 450, 650, 300, and 0 Oe during a field increasing and decreasing cycle. It is clear that the magnetic structure shown in Fig. 1 is remarkably different from the smooth flux penetration patterns expected from the usual critical state of a type II superconductor. Instead, magnetic flux penetrates into the MgB_2 films in a pattern like tree-branch. At ~600 Oe, full penetration was reached, as shown in Fig. 1(d). When the external field is subsequently reduced (Figs. 1(e) and (f)), flux at some locations of the film edge exits at an abrupt manner. Furthermore, as the external field approaches zero, antiflux (the one with opposite polarity) forms near the edge due to the penetration of the reverse return field of the trapped vortices. The combination of the trapped flux of initial polarity and the antiflux can be easily seen in the Fig. 1(f) showing the remnant state $(H = 0)$.

B. Flux Jumps in the c-axis Oriented MgB_2 Thin Film

In some parts of the film, we also observed the abrupt flux penetration into zero-field-cooled MgB_2 films by dendritic magnetic structures at low temperature, as shown in Fig. 2(a). The same kind of peculiar dendritic flux jump has been reported earlier by Johansen and co-workers on the similar films at low temperature [6]. However, we found that this dendritic flux pattern did not necessarily occupy the entire films at temperatures as low as 4.2 K, which is different from what was observed by Johansen and co-workers. In fact, both dendritic flux penetration (Fig. 2(a)) and tree-like flux penetration (Fig. 1) can co-exist in the same film at low temperatures.

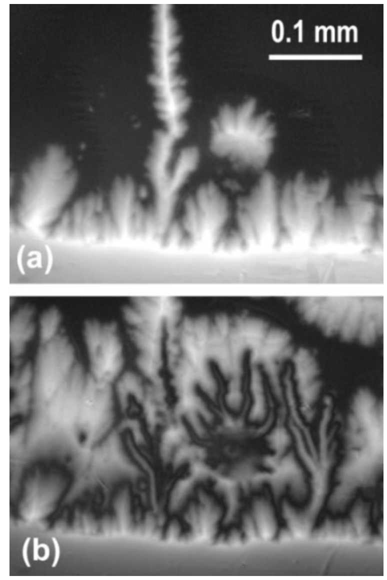

Fig. 2. MO images of the dendritic flux jump in MgB_2 film at 5.5 K. (a) Dendritic flux penetration into zero-field-cooled film at $H = 150$ Oe; (b) Remnant state after external field decreased from 650 Oe to zero, showing the nucleated dendritic antiflux.

Fig. 2(b) shows the MO image of the dendritic magnetic structure at the remnant state after the external field was decreased from 650 Oe to zero. When decreasing the external field, we also observed the exiting flux jump in the dendritic flux structure, and formation of a similar shaped dendritic antiflux at the same time.

At temperatures higher than ~10 K, No flux jump was observed with the dendritic magnetic structure. This is consistent with the results reported by Johansen and co-workers. As temperature approaches T_c, the tree-like magnetic structure gets smeared as expected.

The temperature range where flux jump was observed are found to be the same as that where sudden change was observed in the bulk magnetization of the same film in the hysteresis $(M–H)$ loop measurements. This flux jump is attributed to the thermo-magnetic instability of the flux dynamics in high J_c MgB_2 samples. The heat released by the moving vortices results in a temperature rise (particularly at low temperature where specific heat is low), which further induce vortex motion, and trigger a local avalanche in the critical state. It is clear that thermo-magnetic stability will be a crucial issue for the practical application of MgB_2. Baziljevich and co-workers found that the presence of a heat sink, like a thermally conducting foil, inhibits the formation of the observed dendritic pattern [9].

C. Strongly-Coupled Grain Boundaries in the MgB_2/Mg Nano-Composites

To increase the thermo-magnetic stability of bulk MgB_2, we prepared high J_c MgB_2/Mg nano-composites. It is expected that ~25$\%$ addition of Mg in the MgB_2 matrix will substantially help the heat dissipation generated by the vortex motion, and thus prevent the critical state avalanche. Direct transport and

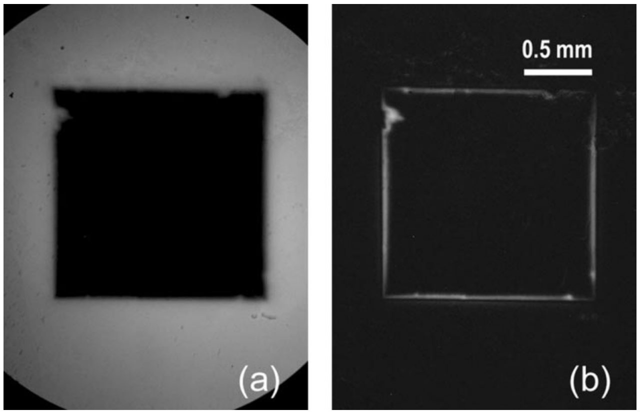

Fig. 3. MO images of the same cross-section of a square disk of MgB_2/Mg nano-composites at 4.2 K. (a) Flux screening at maximum field of 1100 Oe, applied to the zero-field-cooled samples. (b) Remnant state showing surface pinning. Similar patterns were observed at temperatures from 4.2 K to 25 K.

bulk remnant magnetization measurements of these bulk composites show that the grain boundaries are strongly coupled.

Fig. 3 shows the MO images of the same area of a square-disk-shaped specimen cut from the bulk MgB_2/Mg nano-composites without polishing. The broad section (1.5 mm $\times$ 1.5 mm) of the specimen was used for imaging. The cutting by the diamond saw introduced a few microcracks and rough edges into the specimen, which was considered during the image analysis. Fig. 3(a) is the image taken at 4.2 K and at the maximum field of 1100 Oe applied to the zero-field-cooled samples. We found that external magnetic field is completely screened out. Fig. 3(b) is the remnant state after the external field was decreased from 1100 Oe to zero. Brightness intensity analysis of the MO image indicated that there is no penetration or trapping of field inside the specimen. Instead, only surface pinning of magnetic flux was observed. Similar MO images were obtained at 4.2 K $\leq T \leq$ 25 K.

D. Flux Penetration in the MgB_2/Mg Nano-Composites at Temperatures Near T_c

At low temperatures, we were unable to observe the full flux penetration. Bulk magnetization measurements showed that the full penetration field at 4. 2 K was over 7000 Oe. The maximum field supplied by our MO set up is limited to 2000 Oe. Only at $T \geq 35$ K did we observed the full flux penetration at the external field of 1100 Oe.

Fig. 4 is a sequence of MO images taken during a field cycle at 32 K, showing partial flux penetration and trapping into a zero-field-cooled square-shaped MgB_2/Mg specimen. Fig. 4(c) shows the regular flux distribution at highest applied field of 1000 Oe. Fig. 4(c) is not exactly a uniform rooftop distribution pattern of magnetization predicted by the Beam critical state model for a square sample. We believe the discrepancy is due to certain macroscopic defects, e.g., surface notches, introduced during the specimen cutting process. For instance, the excessive flux penetration at lower-left corner shown in Fig. 4(a) is perhaps the evidence of such a kind of surface notch. No indication of electromagnetic granularity was observed on the scale above the MO spatial resolution of 2–5 μm. This is expected because

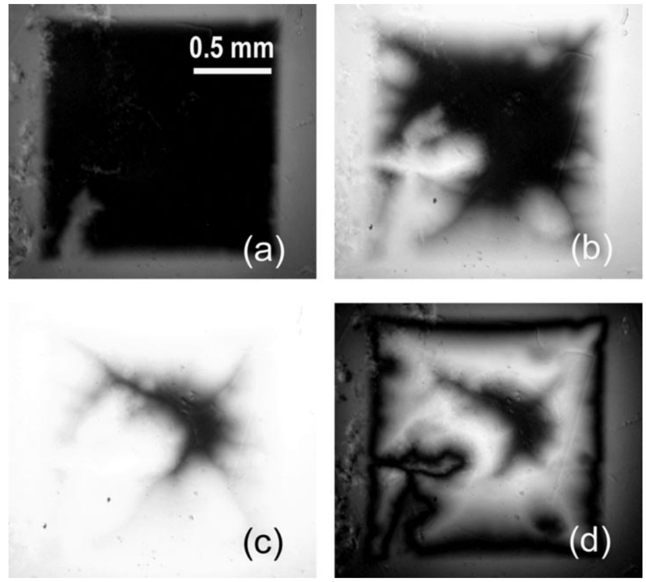

Fig. 4. MO images of flux penetration and trapping in a square disk of zero-field-cooled MgB_2/Mg nano-composites at 32 K. The images were taken in a sequence at $H = 200$, 600, 1000, and 0 Oe during a field increasing and decreasing cycle.

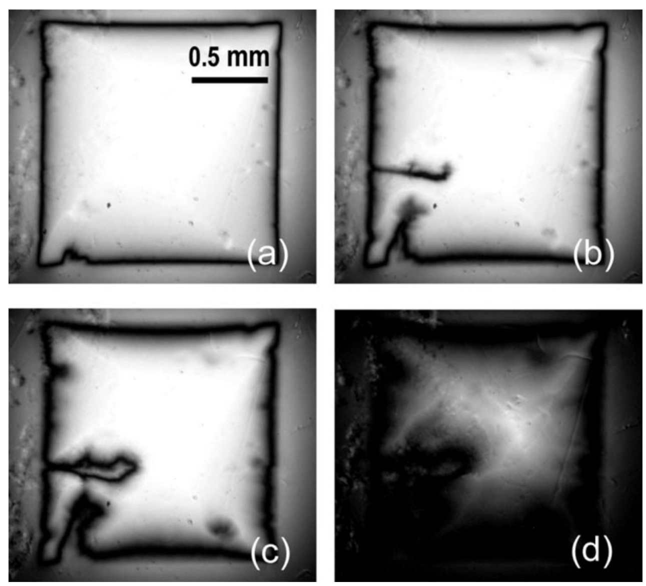

Fig. 5. MO images of flux exit from a square disk of field-cooled MgB_2/Mg nano-composites at various temperatures. The sample was initially cooled under external field of 1000 Oe to 4.2 K, and then magnetic field was turned off. The images were taken at $T = 5.2$ K (a), 25 K (b), 31 K (c), and 33 K (d), respectively, when temperature was gradually increased.

that grain size of this nano-composite is well below the MO resolution. A rather uniform rooftop pattern of flux distribution was reported earlier by Polyanskii and co-workers in polycrystalline MgB_2 [7].

E. Flux Exit From the Remnant State of the MgB_2/Mg Nano-Composites

Fig. 5 is a sequence of MO images showing magnetic flux exit from the remnant state taken at various temperatures from 5.2 K to 33 K. The sample was initially cooled to 4.2 K under external field of 1000 Oe. The distribution of the trapped flux was quite uniform in the specimen up to 20 K. Massive flux exit was observed at $T \geq 31$ K. At 4.2 K $\leq T \leq$ 20 K, the MO images were very stable and essentially the same. In contrast to the case of MgB_2 film, we do not observed any kinds of flux jump behavior at low temperatures, which suggests that addition of Mg in this composite may have indeed prevent the local critical state avalanche, by providing better thermo- and electric-conduction of the composites. It is important to point out the geometric difference between the thin film and bulk specimen in the analysis of the corresponding MO images. For the thin film sample used in this study, the demagnetization factor plays a critical role in the behavior of flux motion and magnetization distribution. The effective magnetic field at the edge of the film could be over an order of magnitude higher than the external field [8]. This huge difference was recognized in making the comparison of MO images on the thin film and the bulk specimen.

IV. Conclusions

In conclusion, we report our comparative MO studies on the high J_c c-axis oriented MgB_2 thin film and the high J_c MgB_2/Mg nano-composites. For the thin film specimen, we observed both dendritic flux jump and tree-like flux penetration patterns in the same film at low temperatures. At $T \geq 11$ K, we only observed a rather stable tree-like flux penetration with no evidence of flux jump in the thin film. For bulk composite specimen, we observed a regular flux distribution, which is consistent with the prediction of a uniform critical state model. No flux jump was observed at temperature as low as 4.2 K even at maximum external field of 1100 Oe. This finding indicated that addition of pure Mg helps to stabilize flux motion and prevent local critical state avalanche. MO images taken at both magnetic field shielding and remnant states suggest that the grain boundaries in this composite are strongly coupled. We also found the MO observations of both the thin film and the composite MgB_2 superconductors are in good agreement with a separate bulk magnetization measurement conducted on the same specimens.

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