# Anomalous Magnetic Hysteresis in the Microwave Surface Resistance of MgB<sub>2</sub> Superconductor

A. Agliolo Gallitto, M. Bonura and M. Li Vigni

CNISM and Dipartimento di Scienze Fisiche ed Astronomiche, Università di Palermo, Via Archirafi 36, I-90123 Palermo, Italy E-mail: marco.bonura@fisica.unipa.it

Abstract - We report on field-induced variations of the microwave surface resistance in samples of  $MgB_2$  produced by different methods. By sweeping the DC magnetic field up and down, we have detected magnetic hysteresis that can be ascribed to the different fluxon density at increasing and decreasing DC fields.in the critical state of the fluxon lattice. The hysteresis observed in the bulk samples has an unusual shape, which cannot be explained in the framework of the critical-state models.

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# I. INTRODUCTION

The *H*-*T* phase diagram of type-II superconductors (SC) is characterized by the presence of the irreversibility line,  $H_{irr}(T)$ , below which the magnetic properties of the SC become irreversible [1]. The application of a DC magnetic field,  $H_0$ , smaller than  $H_{irr}(T)$ develops a critical state of the fluxon lattice, characterized by the critical current density,  $J_c$  [2,3]. The main consequence of the critical-state effects is the hysteretic behaviour of the magnetization curve, which gives rise to hysteresis in all of the properties involving the presence of fluxons.

Fluxon dynamics can be conveniently investigated by measuring the microwave (mw) surface resistance,  $R_s$ , which is proportional to the mw energy losses [4,5]. Indeed, the variations of  $R_s$ , induced by magnetic fields higher than the first penetration field, are due to the presence and motion of fluxons within the mw-field penetration depth. Most models for the field-induced mw losses [4,5,6,7] assume an uniform distribution of fluxons in the sample, disregarding the critical-state effects. Very recently, we have investigated, both experimentally and theoretically, the field-induced variations of  $R_s$  in SC in the critical state [8] and have accounted for the magnetic hysteresis in the  $R_s(H_0)$  curves of Nb samples, powder and bulk.

Here, we report on the magnetic-field-induced variations of  $R_s$  in samples of MgB<sub>2</sub>, produced by different methods. In the  $R_s(H_0)$  curve, we have detected a magnetic hysteresis that can be ascribed to the critical-state effects. The results obtained in bulk samples show a magnetic hysteresis of anomalous shape, which cannot be explained in the framework of critical-state models.

#### **II. SAMPLES AND EXPERIMENTAL APPARATUS**

The field-induced variations of  $R_s$  have been studied in four samples of MgB<sub>2</sub>, three bulk samples (labelled as B) and one powdered (P). Samples B1 and B2 have been prepared by the one-step method [10]; in particular B1 has been obtained using <sup>11</sup>B and B2 using <sup>10</sup>B. Sample B3 has been produced by the reactive liquid Mg infiltration in <sup>10</sup>B powder [11]. Sample P consists of 5~mg of Alpha-Aesar MgB<sub>2</sub> powder, with grain mean diameter of  $\approx$  100 µm.

The mw surface resistance is measured by the cavity-perturbation technique [4]. A copper cavity of cylindrical shape, with gold-plated walls, is tuned in the TE<sub>011</sub> mode resonating at  $\omega/2\pi \approx 9.6$  GHz. The sample is located inside the cavity, in the region in which the mw magnetic field is maximum. The cavity is placed between the poles of an electromagnet, which generates DC magnetic fields up to  $\mu_0 H_0 \approx 1$  T. Two additional coils, independently fed, permit us to compensate the residual magnetic field. The sample and the field geometry are shown in Figure 1(a). In the mixed state, the induced mw current causes a tilt motion of the whole vortex lattice [7]; Figure 1(b) schematically shows the motion of a flux line, induced by the Lorentz force,  $F_L$ . The mw surface resistance of the sample is determined measuring the sample-induced variation of the quality factor of the cavity using the hp-8719D Network Analyzer.



**Fig. 1.** (a) Field and current geometry at the surface of the sample. (b) Schematic representation of the motion of a flux line.

# **III. EXPERIMENTAL RESULTS**

The field-induced variations of  $R_s$  have been investigated at fixed temperatures when increasing and decreasing the DC magnetic field  $H_0$ . In all of the investigated samples we have observed a magnetic hysteresis in a wide range of temperatures, up to a few degrees below  $T_c$ . In this paper we report only on the results obtained at low temperatures, where the critical-state effects are more significant. All measurements have been performed in zero-field-cooled samples.

Figure 2 shows the field-induced variations of  $R_s$ ,  $\Delta R_s(H_0)$ , obtained at T = 4.2 K by sweeping  $H_0$  from 0 up to 1 T and back down, for the four samples.  $\Delta R_s(H_0) \equiv R_s(H_0) - R_{res}$ , where  $R_{res}$  is the residual mw surface resistance at  $H_0 = 0$ ; The data shown are normalized to the maximum variation,  $\Delta R_s^{max} \equiv R_n - R_{res}$ , where  $R_n$  is the surface resistance in the normal state, at  $T \approx 40$  K. Open symbols refer to the results obtained in increasing fields, full symbols those in decreasing fields. In decreasing  $H_0$ , after it had reached ~1T, we observe an initial reversible behaviour followed by a hysteretic behaviour below a certain value of  $H_0$ , depending on the sample, indicated in the figure as H'.

In all of the investigated samples, the application of a DC magnetic field of  $\approx 1$  tesla, much smaller than the upper critical field, induces an unusually enhanced  $R_s$  variation; dependent on the sample, it ranges from  $\approx 20\%$  to  $\approx 40\%$  of the maximum variation (achieved when the sample reaches the normal state). The enhanced  $R_s$  variation has been ascribed to the unusual fluxon structure in MgB<sub>2</sub>, due to the two superconducting gaps [12,13].

In this paper, we discuss the irreversible properties of  $R_s$  in MgB<sub>2</sub>. A comparison of results obtained in the different samples, shown in Figure 2, indicates that in all of the bulk samples the width of the hysteresis loop at  $H_0 = 0$  is about 50% of the total  $R_s$ variation between  $\mu_0 H_0 = 0$  and 1 T, while in the powder sample it is only  $\approx 10\%$ . Another peculiarity distinguishing the mw responses of the powder and the bulk samples is the shape of the decreasing-field branch of the  $R_s(H_0)$  curve. Indeed, one can see that in the powder sample the decreasing-field branch is nearly linear between  $H_0 =$ H' and  $H_0 = 0$ . On the contrary, in the bulk samples the decreasing-field branch of the  $R_s(H_0)$  curve shows an unexpected plateau extending from  $\mu_0 H_0 \approx 0.2$  T down to 0.



**Fig. 2.** Field-induced variations of  $R_s$ , at T = 4.2 K, for the four samples;  $\Delta R_s(H_0) \equiv R_s(H_0) - R_{res}$ , where  $R_{res}$  is the residual mw surface resistance  $H_0 = 0$ ;  $\Delta R_s^{max} \equiv R_n - R_{res}$ , where  $R_n$  is the surface resistance in the normal state.

The field-induced variations of  $R_s$  have been also investigated by cycling the DC magnetic field in different ranges. Figures 3(a) and (b) show the  $R_s(H_0)$  curves, obtained for samples B2 and P, respectively, by sweeping  $H_0$  from zero up to a certain value,  $H_{\text{max}}$ , and back, for different values of  $H_{\text{max}}$ . As expected, due to the different trapped flux, at smaller  $H_{\text{max}}$  the hysteresis width is smaller. In sample P, we have not reported results obtained with  $H_{\text{max}}$  larger than the H' value of Figure 2(d) because they exactly reproduce those of this Figure. When  $H_{\text{max}} < H'$  the  $R_s(H_0)$  curve is irreversible in the whole field range swept. On the contrary, in the bulk sample the value of  $H_0$  below which the decreasing-field branch deviates from the increasing-field one depends on  $H_{\text{max}}$ . For  $\mu_0 H_{\text{max}} = 0.2$  T, the hysteresis is visible in a restricted range of fields and the decreasing-field branch shows a monotonic decrease. However, for  $\mu_0 H_{\text{max}} > 0.2$  T the unexpected plateau at low magnetic fields is well visible. Similar results have been obtained in all of the bulk MgB<sub>2</sub> samples investigated.



**Fig. 3.**  $R_s$  ( $H_0$ ) curves, obtained for samples B2 (a) and P (b), by sweeping  $H_0$  up to a certain value,  $H_{\text{max}}$ , and back, for different values of  $H_{\text{max}}$ ; T=4.2 K.

#### **III. DISCUSSION**

It is well known that the magnetic-field-induced variations of the mw surface resistance are due to the presence and motion of fluxons within the mw-field penetration depth [4,5,6,7]. In particular, at low temperatures and for applied magnetic fields sufficiently below the upper critical field, these variations are essentially due to the vortex motion induced by the mw current. In most of the models of fluxon dynamics reported in the literature, the fluxon distribution is assumed uniform, neglecting the effects of the critical state of the fluxon lattice. However, a magnetic hysteresis in the  $R_s(H_0)$  curve is expected in the critical state, because of the different fluxon density at increasing and decreasing DC fields. In order to account for this hysteretic behaviour, it is essential to consider the fluxon distribution in the sample determined by the critical current density  $J_c$ .

Recently, we have investigated the field dependence of the mw surface resistance of SC in the critical state [8,9], by taking into due account the fluxon distribution, and we have quantitatively justified the hysteretic behaviour of the  $R_s(H_0)$ curve detected in Nb samples [9]. The field geometry we have used is particularly convenient to investigate such effects, essentially for two reasons. First, the effects of the non-uniform fluxon distribution on  $R_s$  are enhanced, because in the sample surfaces normal to the external magnetic field the mw current, penetrating along the fluxon axis within the mw-field penetration depth, bends the end segments of all the fluxons. Moreover, just in this case, one can calculate the average value of  $R_s$  by integration over the sample surfaces that contribute to the mw energy losses. We have shown that the parameter that mainly determines the features of the  $R_s(H_0)$  curve is the full penetration field,  $H^*$ . In particular, the hysteresis width is related to the value of  $H^*$ ; samples of small size and/or small  $J_c$  are expected to exhibit weak hysteretic behaviour.  $H^*$ determines also the shape of the hysteresis loop. On increasing  $H_0$  from 0 up to  $H^*$ , more and more sample regions contribute to the mw losses, giving rise to a positive curvature of the increasing-field branch of the  $R_s(H_0)$  curve. For  $H_0 > H^*$ , in the whole sample the local magnetic induction depends about linearly on the external magnetic field and the increasing-field branch is expected to have a negative concavity. The shape of the decreasing-field branch is strictly related to the shape of the magnetization curve; it should exhibit a negative concavity, with a monotonic reduction of  $R_s$  in the whole field range swept.

A comparison between results presented in different panels of Figure 2 shows that in the powder sample the hysteresis width is smaller than that observed in the bulk samples. In the framework of the critical-state model, this finding is ascribed to the small value of  $H^{*}$  due to the small size of the powder grains. Furthermore, as expected, the decreasing-field branch of the  $R_s(H_0)$  curve of sample P exhibits a monotonic decrease, starting from  $H_0 = H'$  down to  $H_0 = 0$ . On the contrary, in all of the bulk samples the shape of the hysteresis shows several anomalies. The most unexpected behaviour concerns the plateau observed in the decreasing-field branch of the  $R_s(H_0)$ curve, after the sample had been exposed to relatively high fields. The presence of this plateau is puzzling, because it would suggest that the trapped flux does not change anymore on decreasing  $H_0$  below a certain threshold value, of the order of 0.1 T. A further anomaly is visible in the increasing-field branch, which exhibits negative concavity in the whole field range swept, although the estimated value of  $H^*$  for the bulk samples is few tesla [13]. On the contrary, in the  $R_s(H_0)$  curve of sample P one can note a change of concavity in the increasing-field branch at  $\mu_0 H_0 \sim 0.1$  T, which is a reasonable value of  $H^*$  for such powder sample.

We would like to remark that the value of the magnetic field, at which the decreasing-field branch of the  $R_s(H_0)$  curve deviates from the increasing-field branch could differ from  $H_{irr}(T)$  deduced from magnetization measurements. Indeed, it has been shown that, in samples of finite dimensions, the application of AC magnetic fields normal to the DC field may induce the fluxon lattice to relax toward an uniform flux distribution [14] thus reducing the value of  $H_{irr}(T)$ . The process is particularly relevant for thin samples and/or low critical current. Furthermore, considering the sensitivity of our experimental apparatus, we expect to detect hysteresis for  $J_c$  greater than ~ 10<sup>4</sup> A/cm<sup>2</sup>. In this framework, one can justify the reduced value of H' we obtained in

sample P, as shown in Figure 2(d), which is roughly one order of magnitude smaller than the value estimated using  $J_c$  values reported in the literature for MgB<sub>2</sub>.

The same justification cannot be given for the results obtained in the bulk samples. From Figure 3(a) one can see that in the bulk sample, after the reversal of the field-sweep direction, the  $R_s(H_0)$  curve shows an initially reversible behaviour, independently of the value of  $H_{\text{max}}$ . Therefore, the value of the magnetic field at which the decreasing-field branch deviates from the increasing-field one depends on  $H_{\text{max}}$ . We would like to remark that this result has been obtained in all of the bulk samples we have investigated. This finding disagrees with the meaning of the irreversibility line. Indeed, the region of the *H*-*T* plane in which the superconductor exhibits irreversible magnetic properties is not expected to depend on the magnetic history of the sample, but only on the  $J_c$  value at the corresponding temperature and external magnetic field. This anomaly has not been observed in the powder sample. Indeed, Figure 3(b) shows that when the direction of magnetic field sweep is reversed at fields smaller than the value of H' deduced from Figure 1(d) the  $R_s(H_0)$  curve is irreversible in the whole field range swept, consistently with the expected behaviour.

In the framework of the critical state, the main property distinguishing the magnetic response of samples having small or large dimensions is the residual magnetic induction. Let us consider two samples with the same value of  $J_c$  and different sizes, small and large. Furthermore, let us indicate as  $H^*_{\text{small}}$  and  $H^*_{\text{large}}$  the full penetration field of the small- and large-size sample. In the critical state à la Bean, after cycling  $H_0$  in the range  $0 \rightarrow H_{\text{max}} \rightarrow 0$ , with  $H^*_{\text{small}} < H_{\text{max}} < H^*_{\text{large}}$ , the maximum value of the local magnetic induction inside the samples will be  $B_{\text{max}} = 0.5 \ \mu_0 H^*_{\text{small}}$  in the small-size sample and  $B_{\text{max}} = 0.5 \ \mu_0 H_{\text{max}}$  in the large-size sample. This means that in the regions of the sample far from the edges, the local magnetic field in the large-size sample can be much higher than that in the small-size one. Since the dimensions of our bulk samples are a factor of  $\approx 20$  greater than the grain dimensions of sample P, one may infer that the different behaviour of the irreversible properties of  $R_s$  in the powder and bulk samples, after they had been exposed to relatively high magnetic fields, is related to the high value of the magnetic induction reached inside the bulk samples far from the edges.

# **IV. CONCLUSION**

We have reported on the irreversible properties of the field-induced variations of the microwave surface resistance in different ceramic MgB<sub>2</sub> samples, powder and bulk, produced by different techniques. The results have been qualitatively discussed in the framework of models reported in the literature. The hysteretic magnetic behaviour of  $R_s$  observed in the bulk samples exhibits several anomalies not yet understood. In particular, the magnetic field at which the decreasing-field branch deviates from the increasing-field one depends on the maximum magnetic field reached; furthermore, the decreasing-field branch of the  $R_s(H_0)$  curve shows an unexpected plateau extending from a certain value of  $H_0$ , depending on  $H_{max}$ , down to zero. In the powder sample, consisting of MgB<sub>2</sub> grains of small dimensions, the shape of the observed magnetic hysteresis is consistent with that expected using the critical-state model. This finding suggests that the anomalous response of the bulk samples is strictly related to the higher value of the local magnetic field in the interior of the sample, after the applied magnetic field has reached high values.

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