

## Analysis of magnetization relaxation in MgB<sub>2</sub> bulk samples obtained by electric-field assisted sintering

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### ABSTRACT

The relaxation of the irreversible magnetization of MgB<sub>2</sub> bulk samples obtained by electric-field assisted sintering was investigated using the SQUID magnetometry for a magnetic field  $H$  up to 50 kOe applied in zero-field-cooling conditions. We observed a crossover plastic creep at high temperatures  $T$ -elastic creep at low  $T$ , described by  $H \propto T^{-2}$  in the low  $T$  range, which appears to be caused by the macroscopic currents induced in the sample during magnetization measurements. By decreasing  $T$  below this line the determined creep exponent rapidly overcomes the widely accepted theoretical values for elastic (collective) pinning. This behaviour can easily be explained through the occurrence of micro flux jumps, leading to a finite magnetization relaxation rate in the low- $T$  limit.

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### 1. Introduction

Magnesium diboride is considered at present a reliable choice for large scale applications of superconductivity. The critical temperature  $T_c$  of MgB<sub>2</sub> is a record high for simple metallic compounds [1], and the absence of weak-link behaviour opened up the possibility of a new class of low-cost high-performance superconducting materials. One important issue is to attain competitive values for the critical current density  $J_c$ , which is usually controlled by the vortex mobility–pinning relation.

Vortex pinning in MgB<sub>2</sub> single crystals is weak, which is not the case of well compacted polycrystalline specimens and thin films [2]. While  $J_c$  values approaching the pair breaking limit were found in thin films at low  $H$  [3], a  $\delta T_c$  pinning with a small bundle collective (elastic) pinning in a large portion of the vortex phase diagram was proposed for bulk samples in Ref. [4]. It was also reported that for  $H < 8$  kOe a (dislocation free) Bragg-glass state [5] exists in well compacted samples obtained by hot isostatic pressing [6], whereas a grain boundary pinning [where the pinning behaviour of bulk specimens is described by the traditional (plastic) flux–shear mech-

anism] was discussed in Refs. [7,8]. The experimental investigation of the pinning mechanism in MgB<sub>2</sub> is often based on the  $H$  dependence of the normalized pinning force, with the latter extracted from the DC magnetization curves [9].

An essential tool for the study of vortex dynamics in the presence of pinning is the relaxation of the irreversible magnetization. It was shown [10] that the irreversible magnetization of MgB<sub>2</sub> bulk samples depends logarithmically of time, i.e., the flux-creep activation energy would decrease linearly with increasing current density  $J$ , as expected in the framework of the Kim–Anderson model [11]. On the other hand, AC susceptibility measurements performed on similar specimens [12] revealed the fact that the flux-creep activation energy is a nonlinear function on  $J$ . The relaxation of the irreversible magnetization in the low- $T$  domain suggests that the flux dynamics in MgB<sub>2</sub> may be dominated by quantum effects [13,14], such as quantum fluctuations and tunnelling, but the magnetization measured at low  $T$  is affected by flux jumps or avalanches [15]. Further investigations are needed to clarify these aspects.

In this work we analyze the relaxation of the irreversible magnetization of compact MgB<sub>2</sub> bulk samples prepared by electric-field assisted sintering. We found a large influence of the macroscopic currents induced in the sample during experiments,

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leading to a crossover plastic creep at high  $T$ -elastic creep at low  $T$ , similar to that observed for  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_{8+\delta}$  single crystals [16] and  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$  films [17]. However, by decreasing  $T$  below the creep-crossover line the determined creep exponent rapidly overcomes the widely accepted theoretical values for elastic (collective) pinning, signalling the presence of micro flux jumps. We suggest that the anomalous behaviour of the magnetization relaxation rate at low  $T$  can be caused by the occurrence of micro flux jumps.

## 2. Experimental

$\text{MgB}_2$  bulk samples with the density higher than 90% of the theoretical density were obtained by electric-field assisted sintering. The main characteristic of this technique is that a pulsed current directly passes through the conductive powder compact. The heat is generated internally, in contrast to the conventional hot pressing, where the heat is provided by external heating elements. This method is currently used to consolidate ceramic, metal and composite powders. Preparation details and the microstructure of the specimens investigated in this work can be found in Ref. [18]. The characteristic sample dimensions were  $\sim 1 \times 1 \times 0.4 \text{ mm}^3$ , with the largest side perpendicular to the direction along which the pressure was applied during the sintering process.

The magnetization  $M$  was measured using a commercial Quantum Design MPMS in the RSO mode, with  $H$  applied in zero-field-cooling conditions and oriented perpendicular to the largest sample side. The onset of the diamagnetic signal for  $H = 10 \text{ Oe}$  occurs at the critical temperature  $T_c \sim 38.5 \text{ K}$ , and the transition width is around 1 K. In the  $(H, T)$  domain considered below  $M$  was identified with the irreversible magnetization, and the magnetization relaxation measurements were performed with the magnet in the persistent mode. The relaxation time  $t$  was taken to be zero when the magnet charging was finished, and the first data point was registered at  $t_1 \sim 100 \text{ s}$ .

## 3. Results and discussion

Fig. 1 (main panel) illustrates the DC magnetization curves  $M(H)$  obtained with the magnet in the hysteresis mode for  $T$  between 2 K and 28 K. One can see the presence of macroscopic flux jumps [19] at  $T = 2 \text{ K}$  and  $T = 5 \text{ K}$  for  $H$  below  $\sim 20 \text{ kOe}$ . The inset to Fig. 1 shows  $|M|$  vs.  $\ln(t)$  for  $H = 30 \text{ kOe}$  at several  $T$  values (where no macroscopic jumps on the DC magnetization curves are seen in our measurements), which would suggest that  $M$  relaxes logarithmically in time, in agreement with the Kim–Anderson model [11]. As known, in the framework of this approach the vortex-creep activation energy  $U(J) = U_0(1 - J/J_c)$ , where the barrier  $U_0$  can depend on  $H$  and  $T$ . The above  $U(J)$  form leads to a logarithmic  $M(t)$  variation,  $M(t) = M(t_0)[1 - (T/U_0)\ln(t/t_0)]$ , where  $|M| \propto J$  and  $t_0$  is the macroscopic time scale for creep (of the order of  $10^{-3} \text{ s}$ ) [20].

In the linear  $U(J)$  model [11] the relaxation data from the inset to Fig. 1 allows the determination of the magnetization relaxation rate [21]  $S_0 = -[\Delta M/M(t_0)]/\Delta \ln(t) = T/U_0$ . Fig. 2 (main panel) shows the  $S_0(T)$  dependence for  $H = 30 \text{ kOe}$  with  $M(t_0) = M(10^{-3} \text{ s})$  obtained by extrapolation, whereas the resulting  $U_0(T) = T/S_0$  is plotted in the inset. The decrease of  $U_0(T)$  with decreasing  $T$  in the low- $T$  domain (see the inset to Fig. 2) is unnatural, since at low  $T$  the characteristic superconducting lengths have a slow variation with  $T$ , and  $U_0$  should not change significantly with  $T$ . (At this point, it is worthy to note that for  $H$  above  $\sim 10 \text{ kOe}$  the contribution of the  $\pi$  band to the superelectron density is negligible [2].) The nonmonotonous  $U_0(T)$  variation appears for every plausible  $t_0$  value.

In order to reduce the recognized intrinsic ambiguity of flux-creep experiments [related to  $M(t_0)$ , for example], it is better to

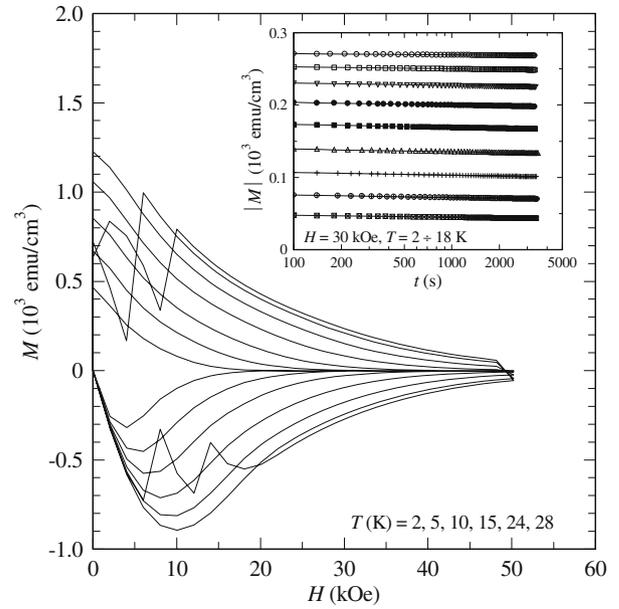


Fig. 1. Main panel: DC magnetization curves  $M(H)$  of compact  $\text{MgB}_2$  bulk samples for several  $T$  values between 2 K and 28 K, obtained with the magnet in the hysteresis mode and the step  $\Delta H = 2 \text{ kOe}$ . For  $T = 2 \text{ K}$  and  $5 \text{ K}$  and  $H$  below  $\sim 20 \text{ kOe}$  macroscopic flux jumps are present. The inset illustrates the absolute value of the magnetization  $|M|$  vs.  $\ln(t)$  for  $H = 30 \text{ kOe}$  and  $T$  between 2 K and 18 K (with the step in  $T$  of 2 K). The weak magnetization relaxation and the usually small relaxation time window are responsible for the fact that  $M(t)$  appears to be linear in  $\ln(t)$ .

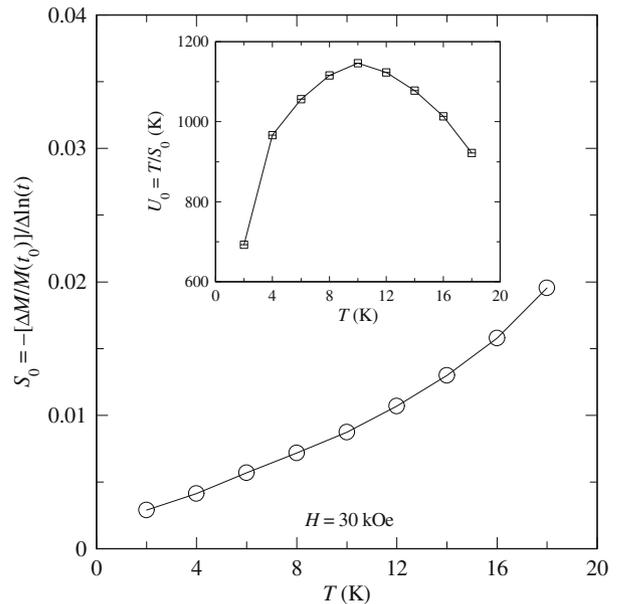


Fig. 2. Main panel:  $T$  variation of the magnetization relaxation rate  $S_0 = -[\Delta M/M(t_0)]/\Delta \ln(t)$  determined using the magnetization relaxation data from the inset to Fig. 1 [ $H = 30 \text{ kOe}$ , and  $M(t_0) = M(10^{-3} \text{ s})$ , obtained by extrapolation]. The resulting pinning barrier  $U_0(T) = T/S_0$  is plotted in the inset.

analyze the  $J$  dependence of the normalized vortex-creep activation energy  $U^* = -T \Delta \ln(t)/\Delta \ln(|M|)$  [21]. [For a limited relaxation time window and a weak  $M(t)$  variation, the instantaneous  $U^*(J) = -T \text{d} \ln(t)/\text{d} \ln(|M|)$  extracted from a single magnetization relaxation curve is highly affected by the measurement sensitivity and the error in setting the moment  $t = 0$ .] Plotting  $\ln|M(t)|$  vs.  $\ln(t)$  the (almost) linear variation is preserved, and we determined a normalized magnetization relaxation rate  $S = -\Delta \ln(|M|)/\Delta \ln(t)$

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