Measurement of Multiple Magnetization Characteristics of High T_c Superconductors

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Abstract

In this paper we present tools for measurement of multiple magnetization characteristics using the compensation techniques and the 2 –order SQUID gradiometric apparatus. The examples of the multiple measurements of AC and virgin DC magnetization M vs. applied magnetic field H_a dependencies of HTc superconductors are shown. Usefulness of the multiple characteristics is discussed.

1. Introduction

AC and DC methods of measurement are commonly used in determining of magnetic properties of materials. The most sensitive instruments available for the magnetic measurements use a superconducting quantum interference device (SQUID) - magnetic flux sensor. The SQUIDs are the most sensitive magnetic flux detectors known today. Their operation is based on magnetic flux quantization and Josephson effects which are consequences of the macroscopic quantum coherence of the superconducting state.

The SQUID system is also ideal for study of the samples where the material characteristics or magnetic effects under investigation require very small excitation fields. In this connection superconductors represent extra significant material type. With respect to the investigation of superconductors in low magnetizing fields, there are some interesting surface or internal structure effects which has not been fully explained up to now, e.g. the paramagnetic Meissner - Ochsenfeld effect, the mechanisms determining a penetration magnetic field into the sample volume or explaining anisotropic behavior of high temperature superconductors.

The fundamental properties of superconductors are zero resistivity and Meissner effect, i.e., magnetic flux expulsion from interior of the sample below the critical temperature T_c, even if the magnetic field was already within the sample before cooling, resulting diamagnetic response on the applied field. Recently, a paramagnetic response (referred as paramagnetic Meissner effect (PME) or also Wohlleben effect) have been reported when certain samples of high-T_c and also low-T_c superconductors were cooled in relatively low fields. Several explanations for the origin of the PME have been given so far [1, 2]. Usual experimental tools for PME study are the temperature dependencies of the magnetization in field cooling conditions. For detailed studies of the PME or probing for some models the multiple magnetization M vs. the applied field H_a characteristics presented in the paper could be particularly interested. Higashitani in [3] proposed that the presence of a large sharp zero-energy peak in the quasi-particle density of the states causes PME. If the magnetic field is applied to a superconductor, then a surface supercurrent is induced. This can be expressed by means of diamagnetic (pair) and paramagnetic (quasi-particle) current components. The multiple M vs. H_a curves for low applied fields could be tools to identify a limit of their significance effects.

The discovery of materials with critical temperature higher than the liquid Nitrogen temperature stimulated important activity for the search of a mechanism of superconductivity of the materials. So far there is no consensus regarding this matter. The common feature of the new family superconductors is their layered structure along the c-axis comprising the existence of sheets of CuO_2 planes. It is now widely accepted that pairs of holes doped in the CuO_2 plane are responsible for the high temperature superconductivity. It is generally believed now that the extremely large anisotropies between the a-b plane and c-axis directions can be explained by intrinsically two dimensional superconductivity in the CuO_2 layers weakly coupled together by the Josephson currents along the c-directions [4]. Hussey et al. in [5] used multiple consecutive low field $M(H_a)$ curves for study and classification of the interlayer coupling in high-T_c superconductors.

Means of measurement of multiple AC and DC magnetization characteristics are presented in this paper and demonstrated on measurement of some high-T_c superconductors.

2. Experimental

The AC and DC volume magnetization characteristics were measured by the compensation method using 2-nd order SQUID gradiometer. The sample is outside the pickup superconducting antenna. It is inserted in the coil set consisting of magnetization, calibration and compensation coils placed outside the helium cryostat, [6]. The antenna is made from the superconducting wire coil systems which are set up as the second order gradiometer. The signal from the antenna is conveyed to the SQUID sensor. The effect of magnetizing field in the gradiometric superconducting antenna is suppressed by the design of coil set and using compensation signal. Detected change in magnetic flux is only due to the changing magnetic moment of the sample as it responds to the changing magnetizing field. So, the detector output is proportional to the change of the magnetic moment and not to the moment itself. The volume magnetization is determined by proper calibration and scaling. Note that no sample movement is required to produce output signal. All magnetization characteristics of the samples were measured at 77.3 K after the zero-field cooling in the magnetic fields ranging from 10^{-1} to 10^5 A m^{-1}. For AC magnetization measurements a frequency of 0.1 Hz has been used. The applied magnetic field was always parallel to cylindrical axis of the sample. The demagnetizing factor was determined from the dimensions of the cylindrical pellet samples [7].

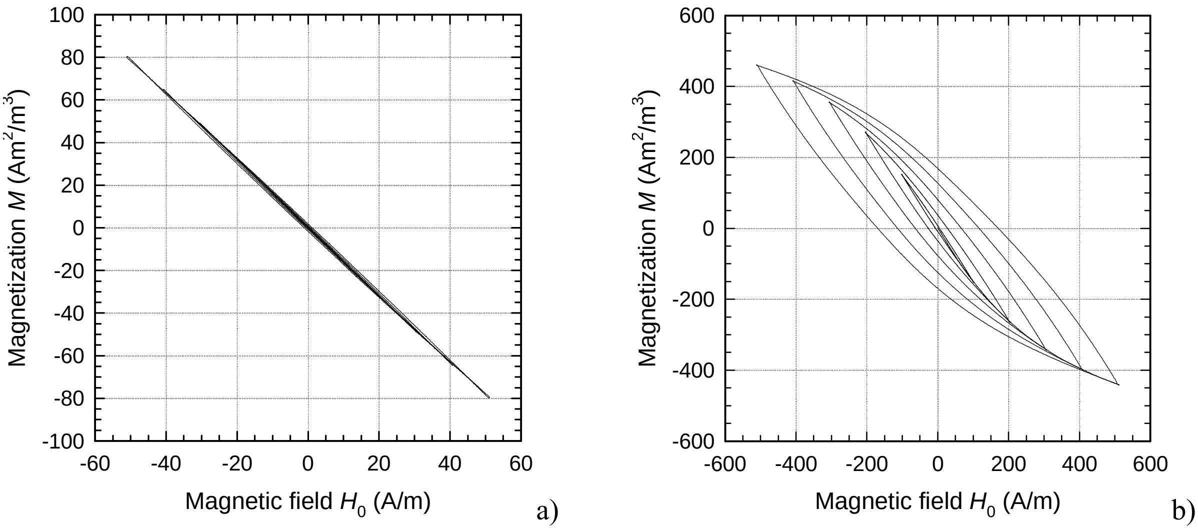
The tested samples were sintered high-T_c (BiPb)-2223 compounds. The samples had a form of cylindrical pellets with a diameter of 10.3 mm and a thickness of 1.6 to 1.9 mm. We scanned the magnetization loops M vs. H_0 for different amplitudes of applied magnetic field H_a (H_0 is corrected applied field H_a by the demagnetization factor) in steps of five amplitudes for each chosen range in order to see more details, such as: the effective magnetic susceptibility in the case of Meissner shielding of the whole sample's volume, the penetration magnetic fields characterizing the onset of the penetration of the magnetic flux into the intergrain weak links and into the intragrains.

3. Results and discussion

Multiple AC magnetization hysteresis loops M vs. the corrected applied magnetic field H_0 of (BiPb)-2223 sample for five magnetization field amplitudes are shown in Figures 1a.-1c. The range of scanning amplitudes of magnetizing field in the Figs. 1a), 1b) and 1c) correspondents to the states of non-hysteretic behavior, starting of magnetic flux penetration into the sample and penetration of magnetic field also into the grains, respectively. The magnetization starting hysteresis loops in figure 1b) resemble the ones of well textured or single crystalline samples without existence of internal structure of shielding current paths. From the shape of magnetization hysteresis loops showed in Figure 1c) the existence of internal structure of shielding currents (the intergrain and the intragrain) can be inferred.

Multiple DC magnetization M vs. the applied field H_a curves of the (BiPb)-2223 sample, are shown in Figures 2a and 2b. The arrows indicate the direction of the field sweep, the arrow (1) belongs to virgin and arrow (2) to the second field sweep. At the first sweep the virgin magnetization M vs. H_a is obtained starting from the zero field cooled state with no trapped magnetic flux (dismissing Earth magnetic field during cooling). The second consecutive field sweep starts from a vortex state inside the sample. The difference between the first and second starting sweeps (at $H_a \sim 0$) is a measure of remanent magnetic moment. An onset of the magnetic field penetration can be determined as the field of first deviation from linear field dependence of the virgin magnetization curve. Using more sensitive derivative techniques, see figures 3a and 3b, the onset penetration field H_p^{on} of about 10 Am^{-1} or after demagnetization correction of 40 Am^{-1} are determined. Figures 3a and 3b show dependence dM/dH vs. H_0 of the multiple $M(H)$ curves shown in Figures 2a and 2b and are scaled that way that dM/dH represents the magnetic susceptibility χ. It is seen that up to H_p^{on} the susceptibility $\chi = -1$, i.e. the whole sample is shielded in a state of ideal diamagnetic Meissner shielding.

In the ref. [5] the multiple low-field M vs. H_a characteristics were used for the study of anisotropy of high-T_c superconductors and to probe the interlayer Josephson couplings in single



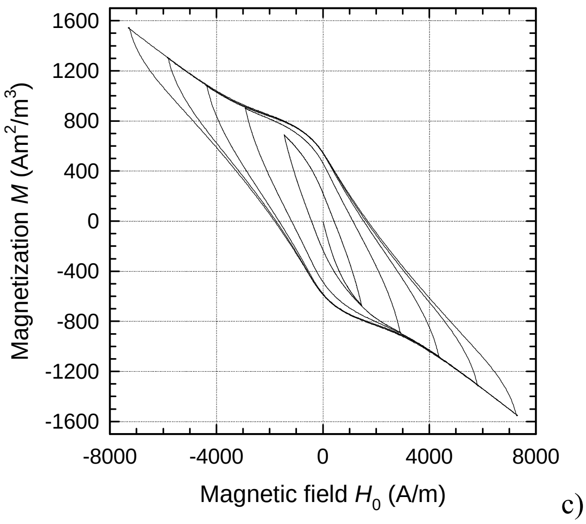


Figure 1. Multiple AC magnetization (M vs. the applied magnetic field H_0 - corrected by demagnetization factor) hysteresis loops of the (BiPb)-2223 sample for five H_0 field amplitudes in the three consecutive growing ranges of the field: a), b), c).

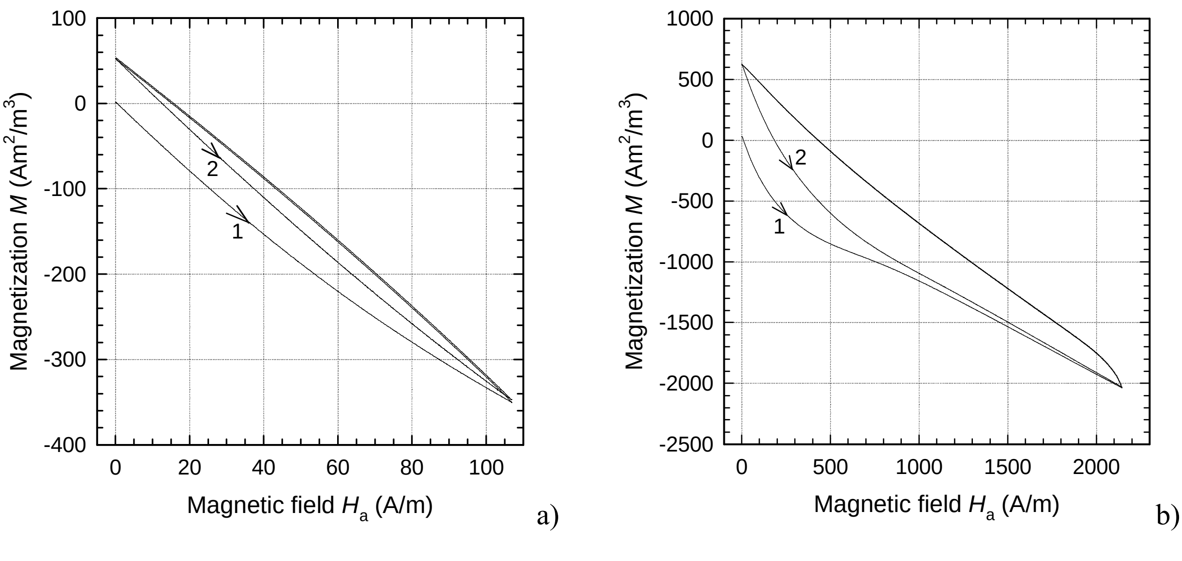
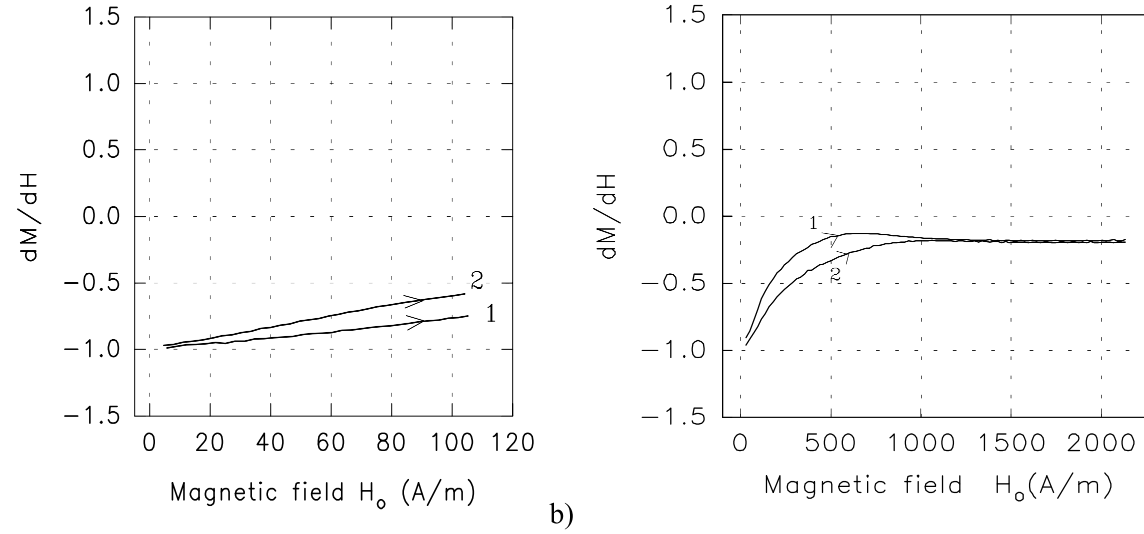


Figure 2. Multiple DC magnetization (M vs. the applied field H_a) curves of the (BiPb)-2223 sample for lower a) and higher b) applied fields. The arrows indicate the direction of the field sweep: the arrow (1) belongs to virgin and arrow (2) to the second field sweep.



a) b)

Figures 3a and 3b. The dependences of derivative dM/dH vs. H_0 of the multiple DC magnetization $M(H_0)$ curves shown in figures 2a and 2b, respectively. The reverse sweep curves were deleted for better lucidity.

crystalline samples. The samples studied in the paper are polycrystalline. Nevertheless, granular superconductor is frequently defined as a material consisting of well superconducting domains - grains connected by the weak superconducting Josephson links. It is known, that the magnetic properties of single crystal or well melt textured samples can be explained by the critical state model (CSM) [8] or extended CSMs [9]. It has been also shown that the critical state model works at least roughly for intergrain matrix of sintered polycrystalline material alone. So, we can speak about the double CSM, [10].

Based on the conception and results of multiple magnetization characteristics of (BiPb)-2223 samples the following can be concluded: at virgin increasing of applied field the sample is field shielded by the ideal paramagnetic Meissner current. If applied field exceeds the onset penetration field 40 Am^{-1} the magnetic vortices penetrate into the intergrain regions and are being pinned. From linear behavior of dM/dH vs. H dependency, see Figure 3a and also partially Figs. 3b and 3c, it can be inferred that the critical state behavior occurs at least in two field ranges, namely when vortices penetrate into intergrain Josephson weak links medium and then when the vortex penetration into the grains starts to dominate.

Acknowledgements

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