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The 2nd harmonic signal analysis in Ca-doped Y-123 superconductors: Sensing DC-fields down to 0.5 nT

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ABSTRACT

We have investigated the DC-field sensing performances of a series of Ca-doped polycrystalline $YBa_2Cu_3O_{7-\delta}$ superconductors. The second harmonic signal generated by applying the AC and DC fields was processed in the designed magnetic sensors. Optimal magnetic sensor properties were achieved on the Ca-doped sample with composition $Y_{0.94}Ca_{0.06}Ba_2Cu_3O_{7-\delta}$. Small DC-fields down to 0.5 nT could be detected with the transfer functions (dV/dB) of 23,300 V/T. The dynamical range of the sensor can be extended up to ~0.5 Oe. The experiments were repeated to test the validity and reproducibility of the results presented.

1. Introduction

Magnetic sensors have been applied in many areas including the underground facilities, hidden weapons, unexploded ordnance, geophysical anomalies, buried foundations, navigation systems and vehicle detection [1,2]. There has been an increased demand for the magnetic sensors to detect small fields in nanotesla and microtesla ranges. Such sensors are required in medical (e.g. imaging organisms) and industrial applications (e.g. non-destructive texturing) [3]. Low field sensors are mainly quantum interference device (SQUID), search coils and fluxgate magnetometers. The SQUID magnetometers may sense fields down to femtotesla, but they work only at liquid helium and possibly at liquid nitrogen temperature. The SQUID magnetometers detect the field gradients rather than the field itself. The search coils on the other hand work in varying magnetic fields or moving through magnetic fields. The search coils are not quite suitable for detecting static or slowly varying fields [4]. The fluxgate magnetometers can measure the magnetic fields down to picotesla and work at room temperature [5–13]. The fluxgate magnetometers are affordable, rugged and compact. These features and low power consumption makes them ideal for a variety of applications requiring low field detection.

In the early 90s, the ceramic superconductors were proposed to develop low field magnetometers. They are based on the same principle as the fluxgate magnetometers but to be used below room temperature [14–16]. In the high-T_c superconductors nonlinear magnetization and low flux entries through the grain boundaries cause the generation of harmonics of the fields [17–21]. In the superconductors the 2nd harmonic signal may be generated by applying AC and DC fields. The linear dependence of the 2nd harmonic signal to the applied DC fields has attracted interests in magnetic sensor technology. Many authors state that weak links between grains of ceramic superconductors are responsible for the sensitivity of the magnetometers [22,23]. On the other hand, our previous studies have shown that such a generalization is quite premature [24,25]. Contrary to the expected improvement, the strength of 2nd harmonic signal appears to increase in the strongly linked grains. In the present study we intend to improve grain boundary links in the $YBa_2Cu_3O_{7-x}$ superconductors by adding calcium atoms. Literature works have shown that the intra-grain critical current density (J_{cg}) drops exponentially if disorientation angle of the grain boundaries exceeds 2–7° [26,27]. It was reported that in the superconductors Ca doping increases the hole concentrations on the grain boundaries and improves J_{cg} [28–31].

This paper presents the effects of Ca doping on the 2nd harmonic signal strength of the superconductor and discusses the sensitivity limit of the magnetometer designed. The optimum Ca concentration and the ways of measuring DC fields lower than 1 nT is reported.

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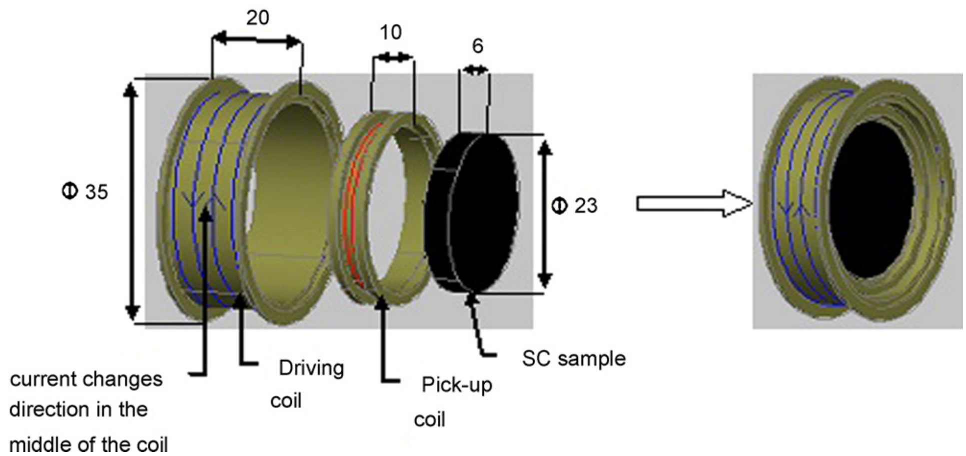

Fig. 1. The coil configuration used in experiments.

2. Experimental

The $YBa_2Cu_{3-x}Ca_xO_{7-y}$ samples with $x = 0.00$, 0.02, 0.04, 0.06, 0.08, 0.1 prepared by the conventional solid state reaction method were used as sensing elements. High purity powders (99.99%) of Y_2O_3, $BaCO_3$, CuO and CaO were weighed in the appropriate amounts and mixed well using an agate mortar. Then, the powder was pressed into pellets under pressure of 1400 kgf/cm^2. The pellets were calcined at 900 °C, 920 °C and 940 °C for 72 h with intermediate grinding, mixing and pelletizing after each 24 h. All the samples were heated at atmospheric pressure and oxygenated under oxygen flow (0.5 l/min) at 940 °C (16 h) and 450 °C (24 h). Then, the samples were sandpapered to have the same dimensions of 23 mm diameter and 7 mm thickness to avoid possible variations arising from demagnetization factor. The M–T and the M–H measurements were done by using MPMS-5 SQUID magnetometer from quantum design. Experimental set up for the second harmonic measurements were described in [24,25]. The pick-up and excitation coils are shown in Fig. 1, the electronics and the test conditions are explained in [24].

3. Results and discussion

3.1. Magnetic measurements

An ideally hard superconductor shows perfect diamagnetism in case the external magnetic field is switched on while the material is in the superconducting state. This is called "zero field cooled" (ZFC) regime. The external magnetic field may be switched on before cooling the sample below the critical temperature. This is called the field cooled (FC) regime. In this case an ideally hard superconductor traps the field in its body and does not allow its exclusion. In practice quite small diamagnetic signal, called Meissner signal may be observed for the FC-mode. Therefore, the difference between the ZFC and FC branches of magnetization curves gives information about the pinning forces. Large difference between the ZFC and FC branches indicate high pinning force and high critical current density. For instance, the difference between the ZFC and FC branches of magnetization is quite large on the melt-textured superconductors and small on the polycrystalline ones. This is due to weak intergranular couplings in the polycrystalline samples. The magnetic flux may easily penetrate through grain boundaries if the grains are weakly connected. Consequently, the M–T measurements on the FC and ZFC modes give information about the nature of the grain boundaries.

The M–T curves of Y-123 samples as a function of Ca concentration are shown in Fig. 2. This figure shows that the onset critical temperature decreases with increase of Ca concentration, in agreement with the published data [31,34,35]. For example, for the sample with $x = 0.10$, the onset critical temperature decreases down to 81 K. However, the transition widths are almost same for all the samples suggesting that the links between grains does not change with Ca addition [36,37]. The trapped fields at the sensor working temperature of 77 K and at 60 K were determined from the M–T curves (see the inset of Fig. 2). The trapped field at 60 K reveals that the pinning force increases with increase of Ca concentration. For instance, at 60 K the trapped field is about 68.2% and 77.1% of applied field for the samples with $x = 0.0$ and $x = 0.1$, respectively. This data indicate that the grain boundary links are improved with Ca addition. This may be associated with the enhanced carrier densities at the Ca-doped boundaries [26–28]. On the other hand, at 77 K the trapped field decreases with increase of Ca concentration. This is an expected behavior since the measurement temperature approaches the transition temperature with increase of Ca concentration. Altogether, the Ca addition improves the grain boundaries, but the pinning force at sensor working temperature (proportionally the critical current density at grain boundaries J_{gb}) decreases with increase of calcium content.

In polycrystalline superconductors, the grain boundary critical field (H_{c1g}) is quite lower than that of intra-grain critical field (H_{c1}). Consequently, the applied field first enters through grain boundaries. The linear variation of the 2nd harmonic signal with DC field is possible for certain cases [32,33]. First, the sum of the AC and DC fields ($H_{dc} + H_{ac}$) must be larger than H_{c1g}. Second, the DC field (H_{dc}) must be quite smaller than the AC field ($H_{dc} << H_{ac}$). Third, the total field must be smaller than the minimum field required for field penetration through the entire specimen (H^*). Therefore, H_{c1g} and the H^* values are important to reach a general notion about the region to work. As seen in Fig. 3, the H_{c1g} and the H^* values in our samples decrease with increase of Ca concentration. The H_{c1g} values are smaller than 1 Oe for the samples having Ca concentration larger than $x = 0.06$. The H^* values decrease down to 30 Oe for the Ca doped sample with $x = 0.10$. Using these values and adjusting the amplitudes of H_{dc} and H_{ac}, the dynamical range of magnetometer can be controlled depending on the area of usage.

Several authors claim that the strength of the 2nd harmonic signal is inversely proportional to the critical current density, J_c ($V_2 \propto 1/J_c$) [38,39]. In order to check the relation of these parameters, The J_c values for our samples were derived from the M–H loops by using the Bean model [40]. The values obtained at 77 K in A/cm^2 unit are, 485, 380, 260, 174, 87 and 73 for the samples with x: 0.00,