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Measurements of ultra-low DC fields by high-T_c superconducting cores: The effect of calcination temperature

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ABSTRACT

In this study, the effects of weak links between grains of the ceramic superconductors on the sensitivity of a DC magnetic field sensor were examined. The evolution of 2nd harmonic signal strength, which determines the magnetometer sensitivity, with critical current density J_c was especially analyzed. The J_c values were adjusted by synthesis of Y-123 superconductors at different calcination temperatures. It was observed that the strength of 2f signal increases as the calcination temperature and so, the J_c values increases. This is contrary to the common expectations. Our experiments have shown that the 2f signal changes quite linearly with DC field in a wide dynamic range (10^{-4} to 10^{-9} T) on the sample calcined at 950 °C. The ways of measuring DC fields as low as 1 nT are described in the paper.

1. Introduction

The magnetic field sensors have a very diverse range of applications from locating submarines, detecting unexploded ordinance to archaeology, planetary exploration and medicine to many more different applications. For most of these applications, sensitivity and reliability have crucial importance. For instance, monitoring of submarine activities requires the detection of magnetic field strength as low as 1 nT (1 nT ~ 8 × 10^{-4} A/m). Together with these applications, rapidly growing interests and developments in nanotechnology have forced metrologists to search new measurement techniques for ultralow physical quantities, such as DC field, by supplying traceability to primary standards. From the magnetic metrology side, constitution of standards for the creation of ultralow DC magnetic fields is relatively easy, but the measurement of such fields is quite difficult. The only known sensitive technique for measuring ultralow DC fields has been fluxgate magnetometry by which DC fields down to several tens of nT can be measured. On the other hand, verification or comparison of the data obtained by such unique methods with other accepted techniques plays a vital role in metrology science; providing confidence in measurements.

A novel form of ultralow field magnetometer, which is based on nonlinear magnetization of polycrystalline type II superconductors, was reported before [1,2]. Many studies have shown that harmonics of reference signal have been generated due to such nonlinear magnetization in case a superconductor is both in AC and DC fields and if the amplitude of total applied field exceeds a threshold value [3–8]. The principle of proposed magnetometer is especially based on the perfect linear variation and the strong low DC field dependency of the second harmonic signal for much small $H_{DC}/H_{AC} \ll 1$ ratios.

In literature, the magnetization of a polycrystalline superconductor is known to be due to the summation of intergranular and intragranular contributions of circulating supercurrents [9]. At low fields intergranular regions dominate the sample's magnetization (i.e. the flux first enters to the specimen through interbrain regions rather than intracranial regions). Therefore, the links between grains are desired to be weak in order to make quite low flux entries possible and so, to increase DC field sensitivity. The amplitude of the second harmonic signal has been expressed with the formula

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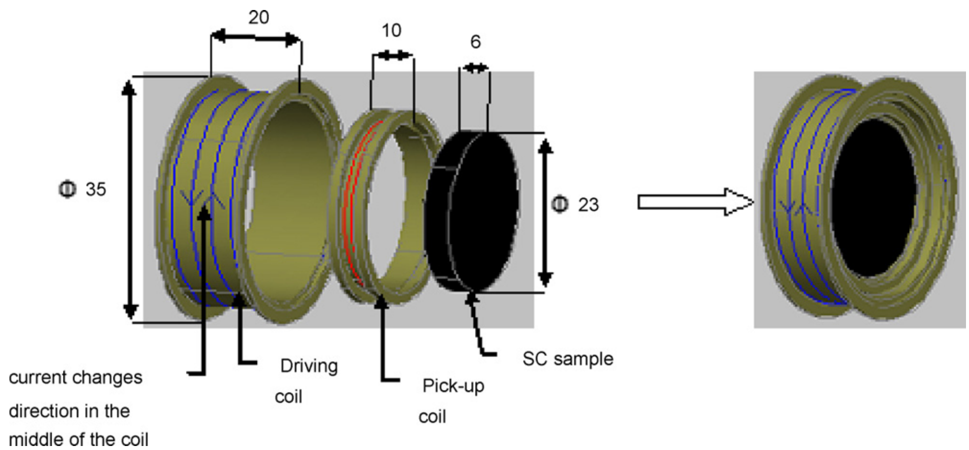

Fig. 1. The coil configurations used in experiments.

of $V_2 \sim \omega H_{DC} H_{AC}^3 AN/J_c$ (where A is the effective area and N is the number of turns of the detection coil) [10,11]. As seen, decrease of J_c (intergranular critical current density) seems to result in the increase of signal strength. On the other hand, there is no systematic experimental data in literature confirming this assumption.

In the present study the polycrystalline $YBa_2Cu_3O_7$ samples were synthesized at different calcination temperatures (@ 900 °C, 925 °C and 950 °C) in order to understand the contribution of intergranular regions to the strength of second harmonic signal. It is known that increasing calcination temperature makes the grain bigger, which causes decreasing of the concentration of weak links and increase of the J_c. Thus, we can get information about the role of weak links on the second harmonic signal and the sensitivity of magnetometer. After clarifying the contribution of weak links we will present the ways of measurements of DC fields lower than 1 nT.

2. Experimental

Experimental set up was explained in Refs. [12,13] in detail. $YBa_2Cu_3O_{7-x}$ (Y-123) ceramic superconductors, used as sensing elements, were prepared by the solid state reaction method. High purity powders (99.99%) of Y_2O_3, $BaCO_3$ and CuO were weighed in the appropriate amounts and mixed well using an agate mortar. Then, the powder was divided into three pieces and pressed into pellets under pressure of 1400 kgf/cm^2. These pellets were calcined at different temperatures (900 °C, 925 °C and 950 °C) for 48 h with intermediate grinding, mixing and pelletizing after each 24 h. Finally, the samples were oxygenated under oxygen flow (0.5 l/min) by holding at 900 °C (16 h) and 450 °C (16 h). The pellets were sandpapered to the same dimensions with diameter of 23 mm and thickness of 6 mm to avoid the variations in demagnetization factor. This size was preferred in order to increase the 2f signal strength. The coil configurations used in experiments are seen in Fig. 1 (the numbers in drawing are mm). The pick-up coil, which was used for the detection of 2nd harmonic signal generated by superconducting specimen, has 2100 turns/cm (wire diameter is 100 μm). The driving coil, which was used for applying an AC magnetic field to the superconductor, has 50 turns/cm (wire diameter is 200 μm) and the winding changes direction in middle of the template. The pick-up coil was moved through the driving coil to minimize mutual inductance between these coils (i.e. mechanical compensation). We must note that the fiber glass was preferred as a template for the coils to minimize thermal expansion in liquid nitrogen.

An electronic filter with high quality factor Q was used to eliminate signals other than 2f = 99.8 kHz (for details, see Ref. [13]). The DC-field was applied by a calibrated solenoid with coil constant of 15.61 Oe/A, which is traceable to primary standards of dimensional and time and frequency over NMR teslameter. The tests were performed at liquid nitrogen temperature and the test place was shielded from surrounding magnetic fields by using trio Mu-metal chambers. The commercial devices used in experiments are mainly; SRS-DS360 ultralow distortion signal generator, Keithley 220 current source, SR844 and SR830 model Lock-in amplifiers and Agilent 54642A model oscilloscope. For electrical connections, well-screened cables were used.

The indentation experiments were conducted with a Berkovich tip using the NH-2 Nanoindenter (manufactured by CETR) with load and displacement resolutions of ±0.1 μN and ±0.03 nm, respectively. Prior to indentation experiments, the samples on the mounting stage were adjusted to prevent tilting in the z-axis. At least five-step load-unload indentation experiments were carried out at different locations of the samples as the nature of the samples was porous and heterogeneous. Loading steps were adjusted as 50, 100, 200, 300, and 400 mN.

3. Results and discussion

3.1. Mechanical measurements

It is well known that the higher the calcination temperature the larger the grain sizes [14]. Consequently, the density of weak links becomes low and the critical current density becomes larger. In literature, it has been reported that superconducting materials having larger grains show mechanically harder characteristics [15]. With this aim, mechanical properties of $YBa_2Cu_3O_7$ samples calcined at different temperatures were examined. It will be also complementary of the magnetic measurements.

Fig. 2 shows that step loading–unloading cycles for polycrystalline YBaCuO samples calcined at 900 °C, 925 °C and 950 °C. The load is cyclically increased from 50 mN up to a maximum of 400 mN. The curves demonstrate that all samples show elasto-plastic behavior during indentation. As can be seen in Fig. 2, different penetration depths were observed for the same peak loads. This implies that the hardness of samples changes with calcination temperature. It is well known that the mechanical properties of high-T_c superconductors exhibit significant and complex variations depending on processing conditions, as well as temperature, microstructure,

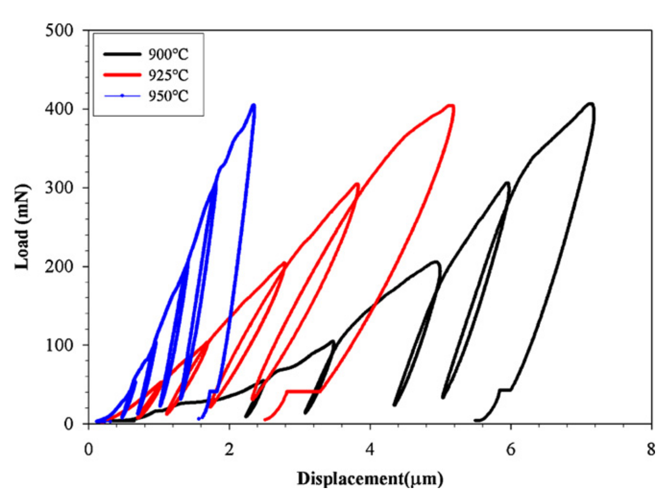

Fig. 2. Step loading–unloading curves of polycrystalline YBaCuO samples calcined at 900 °C, 925 °C and 950 °C.