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Effect of diamagnetic contribution of water on harmonics distribution in a dilute solution of iron oxide nanoparticles measured using high-T_c SQUID magnetometer

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ABSTRACT

The magnetization curve of iron oxide nanoparticles in low-concentration solutions was investigated by a highly sensitive high-T_c superconducting quantum interference device (SQUID) magnetometer. The diamagnetic contribution of water that was used as the carrier liquid was observed in the measured magnetization curves in the high magnetic field region over 100 mT. The effect of the diamagnetic contribution of water on the generation of harmonics during the application of AC and DC magnetic fields was simulated on the basis of measured magnetization curves. Although the diamagnetic effect depends on concentration, a linear relation was observed between the detected harmonics and concentration in the simulated and measured results. The simulation results suggested that improvement could be expected in harmonics generation because of the diamagnetic effect when the iron concentration was lower than 72 μg/ml. The use of second harmonics with an appropriate bias of the DC magnetic field could be utilized for realization of a fast and highly sensitive detection of magnetic nanoparticles in a low-concentration solution.

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1. Introduction

Magnetic nanoparticles have been utilized in medical imaging as contrast agents and tracers [1]. In addition, the use of such nanoparticles in bio-immunoassay, magnetic nanoparticle imaging, and magnetic drug targeting has been studied owing to their promising results in these applications. The inherent magnetic properties of these particles can be determined by measuring their magnetic susceptibility [2–4], relaxation [5–7], and remanence [8,9]. Lately, some researchers have reported the development of sensitive measurement systems that are capable of measuring the magnetic relaxation and remanence of magnetic nanoparticles in solution [7,10,11]. Although the magnetic responses of magnetic nanoparticles in solutions of different concentrations have been widely studied by observing magnetic relaxation, remanence, and AC susceptibility, the behavior of a low-concentration solution of magnetic nanoparticles in wide magnetic field regions still remains unclear. This might be because conventional magnetometers such as low-sensitivity magnetic sensors have certain limitations and the magnetic properties are often measured using concentrated and/or powdered samples. Furthermore, the magnetic response of the nanoparticles is assumed to be similar in solutions of different concentrations and the effect of the diamagnetic carrier liquid is neglected even in diluted solutions. This requires clarification, as most biomedical applications involve measurements of magnetic nanoparticles in low-concentration solutions, wherein the diamagnetic background signal from the carrier liquid may be comparable to the magnetic responses of the nanoparticles. Therefore, development of highly sensitive magnetometers is critical in order to detect small amounts of magnetic nanoparticles in low concentration solutions. Furthermore, magnetic susceptibility must be measured in the presence of an excitation magnetic field, in contrast to the measurement of magnetic relaxation and remanence. Interferences from the excitation magnetic field may limit the sensitivity of measurement systems, particularly in the case of AC susceptometer systems [12–14]. Such interferences from the excitation magnetic fields during dynamic magnetization measurements have been reduced by employing harmonics generated from the nonlinear magnetization characteristics of MNPs [15–17]. These harmonics are used to quantify MNPs with fast measurements and improve their sensitivity by isolating the frequency component of excitation magnetic fields. In addition, large amplitudes of excitation magnetic fields can further lead to enhancement in the generation of harmonics, as they tend

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to cover the wider regions of the nonlinear characteristics.

In this study, we have developed a highly sensitive AC–DC magnetometer using a high-critical-temperature superconducting quantum interference device (high-T_c SQUID) on the basis of our previously developed system [18,19]. This flux transformer-based high-T_c SQUID exhibited high sensitivity with less interference from the excitation magnetic fields. Using the developed system, we measured the static magnetizations and harmonics distribution of low-concentration solutions of iron oxide nanoparticles and investigated their magnetic responses in solutions of different concentrations. As a preliminary analysis to investigate the relationship between the concentration and the harmonics generation of diluted iron oxide nanoparticles during the application of DC and AC magnetic fields, we simulated the generation of harmonics on the basis of measurement results of the static magnetization. The static magnetization curves indicate the contribution of the diamagnetism of water as a function of concentration. Detection of low-concentration magnetic nanoparticles with high sensitivity has been demonstrated using second harmonics.

2. Material and method

2.1. Iron oxide nanoparticles

Iron oxide nanoparticles analyzed in this study were nanomag®-D-spio (Micromod Partikeltechnologie GmbH, Rostock-Warnemuende, Germany). In the typical experiment, a pre-determined quantity of iron oxide nanoparticles was suspended in water such that the concentration of iron in the resulting solution is 2.4 mg/ml. The iron oxide nanoparticles used in this study have an overall diameter of 100 nm, and consist of dextran iron oxide composites. Subsequently, low-concentration solutions were prepared by further diluting the suspension in purified water to obtain iron concentrations of 24 μg/ml, 48 μg/ml, 72 μg/ml, and 96 μg/ml. The diluted iron oxide solutions thus obtained were stored in 3-ml acrylic cases for further analysis.

2.2. AC–DC High-T_c SQUID magnetometer for evaluating magnetic nanoparticles in solution

Fig.1 illustrates the overview of the high-T_c SQUID magnetometer developed in this study and the coil arrangements used for static and dynamic magnetization measurements. The flux transformer consists of first-order planar and axial differential coils as the pickup coils for static and dynamic magnetization measurements, respectively, to reduce the environmental noise being transferred to the high-T_c SQUID. Both types of coils were constructed by connecting two identical elliptical coils in a series opposing configuration. A ramp-edge-type Josephson junction was fabricated on an MgO substrate by a multilayer fabrication technique [20]. The sensitivity of the magnetic susceptibility of the developed system was 1×10^{-8} emu (dimensionless). Details of the measurement system have been reported elsewhere [18,19].

For static magnetization measurements, the sample was exposed to a DC magnetic field generated by an electromagnet and perpendicularly vibrated to the axis of the DC magnetic field using an actuator having amplitude 5 mm and vibration frequency 2.82 Hz. The sensitive axis of the planar differential coil was maintained parallel to the axis of the DC magnetic field, as shown in Fig. 1(a). The induced signals were transferred to the inductively coupled SQUID and the output was detected by a lock-in amplifier. The motion of the actuator that was detected by a laser position sensor was used as a reference signal for the lock-in amplifier. The magnetization curve of the diluted solutions was determined in the range from −260 to 260 mT with a complete cycle of magnetization loop.

The characteristics and the concentration of the iron oxide nanoparticles in the solution could be quantitatively determined from the static magnetization curve; however, this is a time consuming process. Some biomedical applications such as bio-immunoassay require simultaneous measurements of multiple samples. For such applications, harmonics detection [15,21] can provide fast information on the magnetic response of magnetic nanoparticles by covering wide magnetic field regions in one-shot measurements. In this technique, the distribution of the induced harmonics of magnetic nanoparticles is related to the applied AC and DC magnetic field bias and the magnetization curve. For an AC magnetic field with amplitude larger than saturation magnetic field H_s, the magnetic response consists of harmonic components in the region below H_s, whereas in the region above H_s, the magnetic response is suppressed because of moment saturation. However, the introduction of the DC magnetic field will result in the magnetic response of the magnetic nanoparticles following the magnetization curve and production of a distorted waveform of the magnetic field with odd and even numbers of harmonic components [16,17]. Cutting the fundamental frequency component with a filter and measuring the harmonic components of the magnetic response will result in a high signal-to-noise ratio. In our measurement system, the sensitive axis of the axial differential

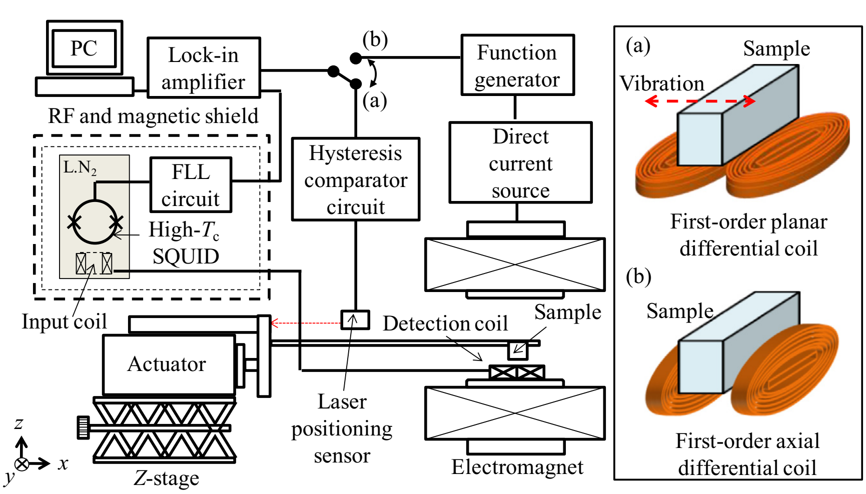

Fig. 1. Schematic diagram of the developed AC–DC magnetometer with coil arrangements of (a) first-order planar differential coil for static magnetization measurement and (b) first-order axial differential coil for dynamic magnetization measurement.

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