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Measurement of metallic contaminants in food with a high-T_c SQUID

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Abstract
We have proposed and demonstrated a high-T_c SQUID system for detecting metallic contaminants in foodstuffs. There is a demand for the development of systems for detecting not only magnetic materials but also non-magnetic materials such as Cu and aluminium in foodstuffs to ensure food safety. The system consists of a SQUID magnetometer, an excitation coil and a permanent magnet. For a non-magnetic sample, an AC magnetic field is applied during detection to induce an eddy current in the sample. For a magnetizable sample, a strong magnetic field is applied to the sample prior to the detection attempt. We were able to detect a stainless steel ball with a diameter of 0.1 mm and a Cu ball less than 1 mm in diameter, for example.

1. Introduction

Recently, processed foodstuffs have become common. Therefore there is a chance of individuals ingesting contaminants which have been accidentally mixed with food. For example, small chips from processing machines and also broken syringe needles used for administering immunization shots or hormone injections, which are mostly metallic materials, have been found in food. In view of the increase in international concern regarding food safety, we need to develop a highly sensitive detector to ensure safety. Although an iron particle detection system has already been developed, there is no system for detecting food contaminants [1–3]. It is difficult to detect all possible contaminant materials with one detection system. However, if targets are limited to metallic contaminants, a high-T_c SQUID is one candidate sensitive detector for finding contaminants in foodstuffs.

In this paper, we describe a system for detecting small amounts of metallic substances by using a high-T_c SQUID magnetometer.

2. Principles

Figure 1 shows the principles of our detection system. Two methods are employed. One is for dealing with non-magnetic samples such as Cu and silver. The other is for dealing with magnetizable samples. For the non-magnetic sample case, an AC magnetic field is applied during detection to induce an eddy current in the sample. The SQUID signal is lock-in amplified and recorded. For the magnetizable sample case, a strong magnetic field is applied to the sample prior to the detection attempt. The direct signal from the SQUID electronics is recorded through a low pass filter, which eliminates the AC field component.

3. Experimental details

3.1. SQUID

The SQUID is made of $Y_1Ba_2Cu_3O_{7-y}$ thin film [4, 5]. The junctions utilized in the SQUID are of the step-edge type. The washer size of the SQUID is about 5.5 × 5.0 mm^2 and the effective area is about 0.1 mm^2. When the SQUID was operated in a flux-locked loop with a flux modulation frequency of 256 kHz, the magnetic flux noise in the white noise region was about 20 $\mu\phi_0$ $Hz^{-1/2}$, as shown in figure 2.

3.2. System

The detection system was located in a magnetically shielded room in our laboratory. Figure 3 shows a schematic drawing of the detection system. A cryostat which was specially designed for use with a SQUID microscope was employed. The cross-sectional view of the cryostat is shown in figure 4. The SQUID was located inside a vacuum, face up, and separated by a 200 μm thick sapphire window. A more detailed description can be found elsewhere [6]. A rectangular shaped Helmholtz coil (1000 turns × 2; bore: 80 mm × 50 mm) was located above the cryostat via an engineering plastic robust table. A sample was slid onto the surface of the rectangular coil tube at constant speed. In this condition the spacing

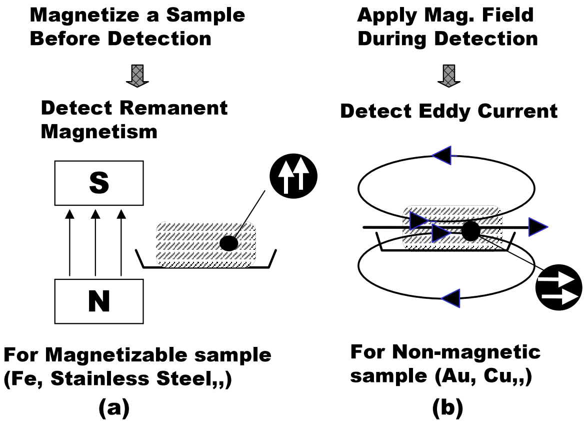

Figure 1. Principles of the detection system. For the non-magnetic sample case, an AC magnetic field is applied during the detection to induce an eddy current in the sample. For the magnetizable sample case, a strong magnetic field is applied to the sample prior to the detection attempt.

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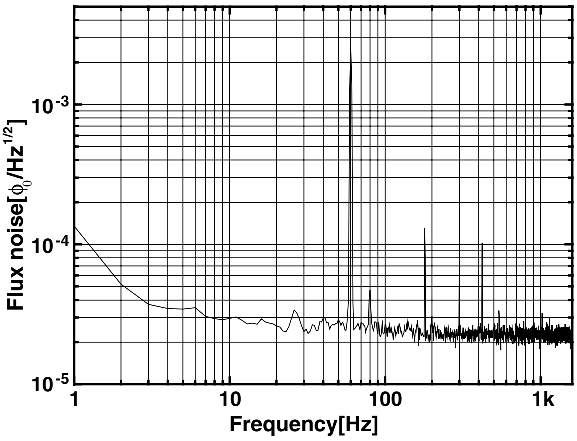

Figure 2. The spectral density of the flux noise of the SQUID. The magnetic flux noise in the white noise region was about $20\ \mu\phi_0\ \mathrm{Hz}^{-1/2}$.

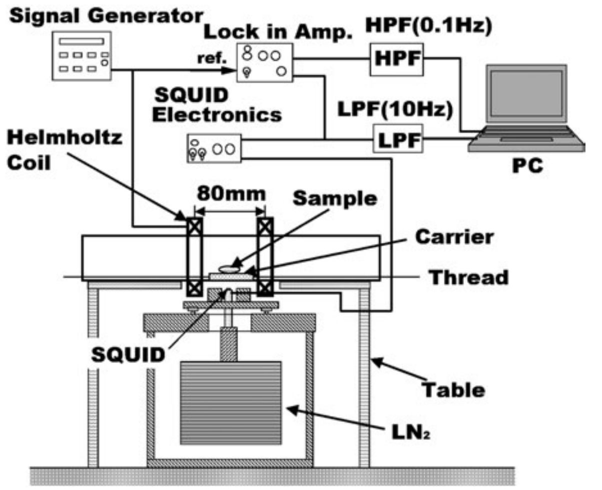

Figure 3. A schematic drawing of the detection system. A rectangular shaped Helmholtz coil was located above the cryostat via a table. A sample was slid onto the surface of the rectangular coil tube with constant speed.

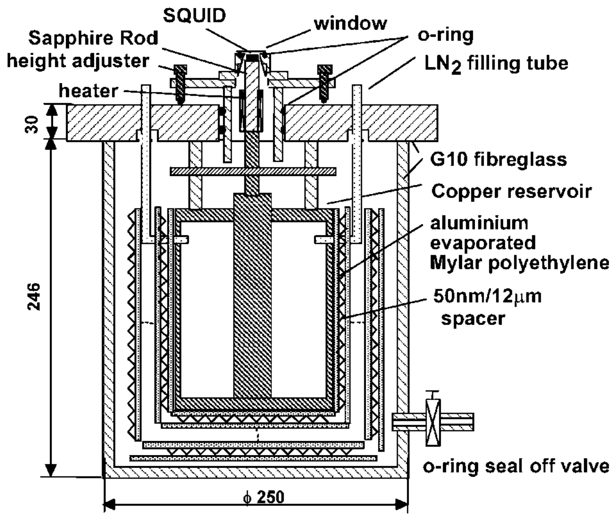

Figure 4. A cross-sectional view of the cryostat. This is especially designed for use with a SQUID microscope. The minimum separation between the SQUID and the sapphire window is 200 μm.

between the sample and the SQUID was from 10 to 50 mm. A sinusoidal AC current with a frequency of 900 Hz–2 kHz was directed to the coils; the peak to peak amplitude of the magnetic field generated from the coil was $2.5 \times 10^{-4}\ \mathrm{T}_{(\mathrm{peak-peak})}$. The modulated signal associated with the metallic sample was then demodulated by the lock-in amplifier. The AC magnetic field was applied to the samples regardless of magnetic properties, such as whether they were non-magnetic or magnetizable samples. For non-magnetic samples, the output signals from the lock-in amplifier were directed to the A/D converter of a PC through a high pass filter with a frequency of 0.1 Hz, which eliminates any dc offset. In this case the z components of the magnetic field at the centre of the coils cancel each other in principle when there is no sample. Therefore the SQUID position was carefully adjusted before measurement, so that the SQUID output signal without a sample became zero [7, 8]. For magnetizable samples, the direct signal from the SQUID electronics is recorded through the low pass filter, which eliminates the AC field component. We note that the samples were magnetized by a 0.5 T permanent magnet prior to the measurements.

3.3. Samples

Austenitic stainless steel balls, stainless steel chips and small copper balls with different sizes were prepared as samples. The size range of the samples was from 0.1 to 1.5 mm in diameter. Since the electric conductivity of austenitic stainless steel is low, it is difficult to detect it using a conventional eddy current method. The austenitic stainless steel material was originally non-magnetic. However, it shows properties like a ferromagnetic material after martensitic transformation during its manufacturing process [9]. Therefore a stainless steel ball sample can be magnetized. So the samples were magnetized for 5 s with a strong permanent magnet before measurement.

4. Results and discussion

Firstly, a stainless steel chip, which was a cut sample of a syringe needle, was measured. It was 0.95 mm in diameter and 1 mm in length. Before the measurement the sample was

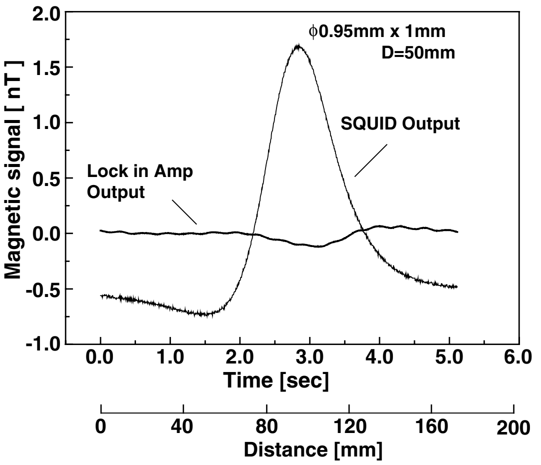

Figure 5. Time domain signal traces for a stainless steel chip. The SQUID output signal shows a large peak: as high as 2.7 nT (peak to peak). The lock-in amplifier output shows a small bump at around the same time.

magnetized with a permanent magnet and then measured. The separation between the sample and the sensor was 50 mm. Time domain signal traces are shown in figure 5, (the second axis at the bottom gives the position of the sample). The SQUID output signal shows a large peak: as high as 2.7 nT (peak to peak). The lock-in amplifier output shows a small bump at 100 mm (2.8 s). This means that the conductivity of the stainless steel is relatively small and as a result the induced eddy current was not large. Meanwhile, it was shown that the sample had a remanent magnetism. Therefore, for the detection of the stainless steel, magnetization is essential to obtain higher sensitivity. Here we calculate the remanent magnetic signal distribution of each sample in order to compare the measured signals. Figure 6 shows the model and the equations used for the calculation. The z component of the magnetic field intensity H_z is a function of r and L, where r is the distance from the dipole to the sensor and L is the length of the dipole. The calculation was performed under the conditions of $r = 50$ mm and $L = 1$ mm. The calculated magnetic field distribution of the sample is shown in figure 7. The measured signal is asymmetric in shape, unlike the calculated signal. The cause of the asymmetry is that the sample was not vertical but slightly tilted. The width of the foot of the measured signal is almost 130 mm, the same as the calculated value. The calculated distribution shows also that the SQUID sensor can cover only a width of 50 mm while keeping a sensitivity of more than 60% of the peak value. This result suggests that multiple SQUID sensors are needed to cover a wide area. This narrow area problem becomes more severe in the case where the sample rotates or tilts upon magnetization.

Secondly, austenitic stainless balls with different sizes were investigated. After magnetization, the sample was slid above the SQUID at a distance of 10 mm. The time signal traces are shown in figure 8. For the diameters 0.6 mm (a) and 0.3 mm (b) of samples, there exists a clear peak at 90 mm (2.6 s). The small dimple at 140 mm (4.2 s) in figure 8(b) is noise coming from the vibration. For the sample of diameter 0.1 mm, a FFT filtering was applied after the data acquisition, because it was hard to identify the peak. The result shown in figure 8(c) is that after filtering. One can identify a small step at around 90 mm (2.6 s) in the figure. The signal peak must be proportional to the volume of the sample. Actually the signal depends on the cube of the diameter. This means that the signals are properly measured. Then we tried to put a sample at some distance from the sensor. Figure 9 shows one of the results for a 0.3 mm stainless steel ball at 30 mm from the sensor. A peak of 0.8 nT was clearly observed at 90 mm (2.6 s). One can compare the peak value with that of figure 8(b). The diameter of the sample is the same. The peak value is inversely proportional to the cube of the distance from the sensor to the sample. Even if the distance became wider than 40 mm, a substantial signal peak could still be detected. The detectable minimum size in the conventional eddy current method for the stainless steel ball is several millimetres. The sensitivity of our system is almost an order of magnitude higher than that of the conventional method. The detectable minimum for our system is adequate for finding contaminants in foodstuffs except for the narrow area problem that we discussed above.

Lastly, we attempted to detect copper ball samples under the modulation scheme. A sinusoidal AC magnetic field was applied along the horizontal direction so that the field did not enter the SQUID. This field induces eddy currents on the surface of the sample. The depth of the current path depends on the frequency. From the calculation, we found that the current flows in bulk for a Cu ball to be less than 1.5 mm in diameter. The distance from the sensor to the sample was 10 mm. The

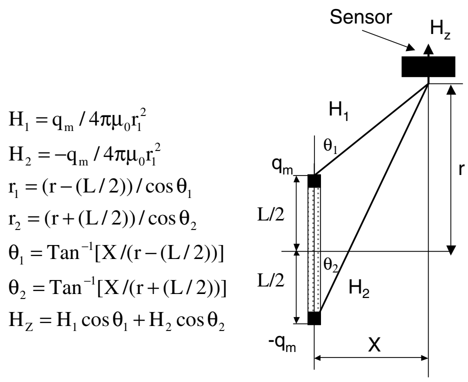

Figure 6. The model and the equations used for the calculation. The z component of the magnetic field intensity H_z is a function of r and L, where r is the distance from the dipole to the sensor and L is the length of the dipole.

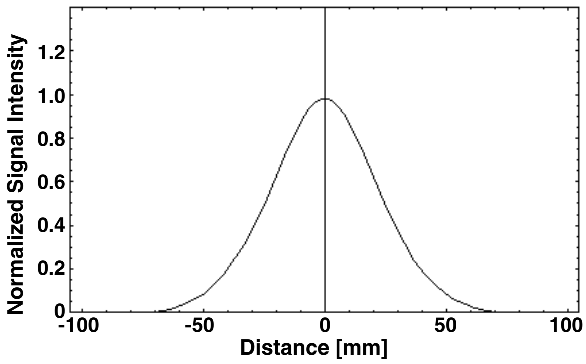

Figure 7. The calculated magnetic field distribution of the sample. The measured signal is asymmetric in shape, unlike the calculated signal, because of the tilt of the sample. The width of the foot of the measured signal is almost 130 mm, the same as the calculated value.

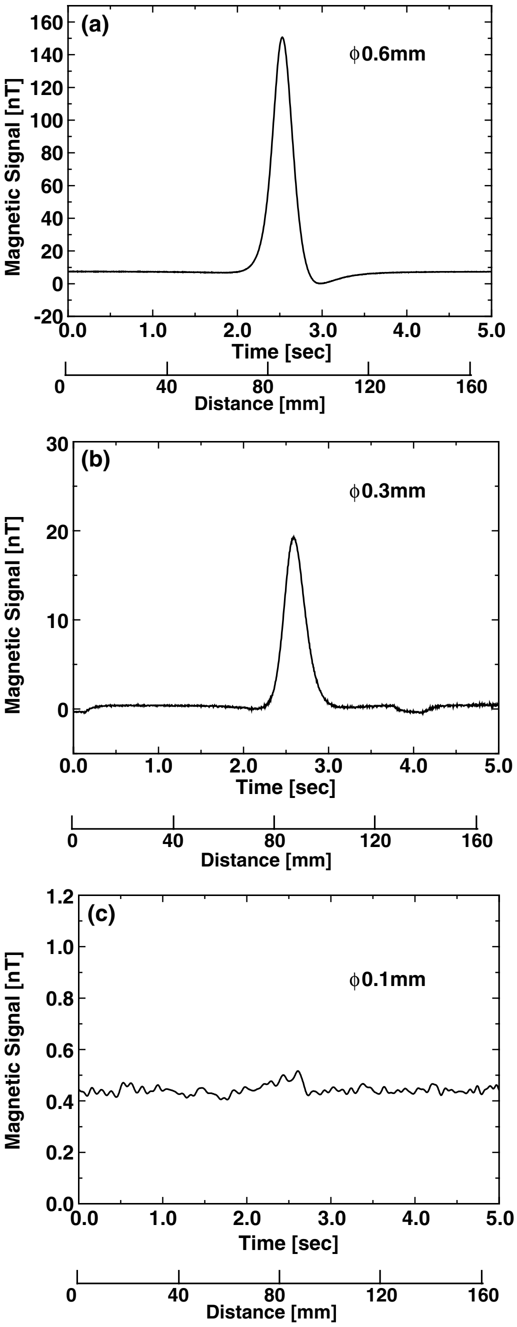

Figure 8. The time signal traces for the diameters (a) 0.6 mm, (b) 0.3 mm and (c) 0.1 mm of samples. For the sample of diameter 0.1 mm, a FFT filtering was applied after the data acquisition.

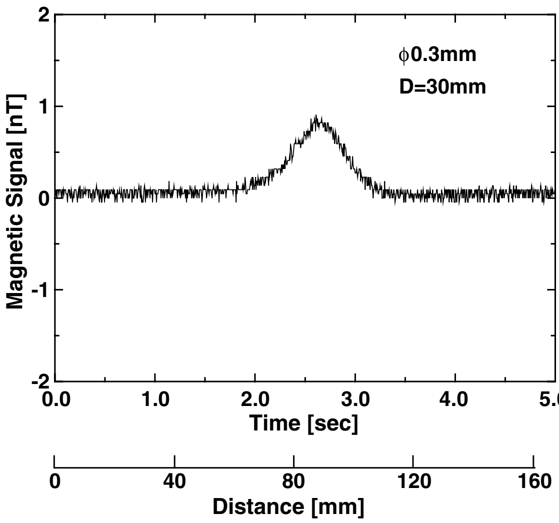

Figure 9. The time signal trace of a 0.3 mm stainless steel ball at 30 mm from the sensor. A peak of 0.9 nT can be clearly observed.

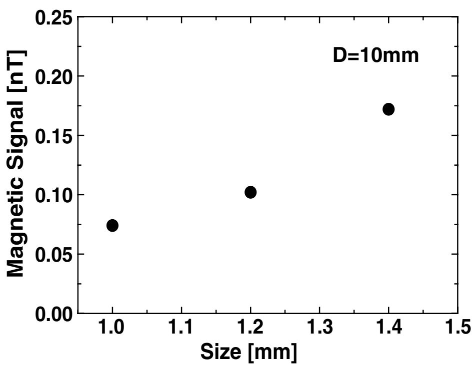

Figure 10. The dependence of the signal intensity on the Cu ball size. At the distance of 10 mm, the detectable minimum size for the Cu balls was 1.0 mm. The signal was proportional to the second to third power of the diameter.

dependence of the signal intensity on the Cu ball size is shown in figure 10. The detectable minimum size for the Cu balls was 1.0 mm. The signals were proportional to the second to third power of the diameter. This suggests that the current follows not only the surface but also the bulk of the sample. We think that the detectable minimum size could be reduced by improving the system so that it can generate a larger AC magnetic field.

5. Conclusion

We have constructed and demonstrated a high-T_c SQUID system for detection of metallic contaminants in foodstuffs. It can successfully detect 0.6–0.1 mm stainless steel balls. The signal showed a good agreement with the calculated value. As regards the detection of non-metallic substances, Cu balls 1.0 mm in size were detected. This detection level is better than market demands. There now needs to be development of a good magnetic shield appropriate to a factory site.

References

[1] Itozaki H 1997 *IEICE Trans. Electron.* **E80-C** 1247–51
[2] Bick M, Sulllivan P, Tilbrook D L, Du J, Thorn B, Binks R, Sharman C, Leslie K E, Hinsch A, Macrae K and Foley C P 2003 *ISEC03: 9th Int. Superconductive Electronics Conf.* PTh06 (Extended abstracts)
[3] Donaldson G B, Cochran A and McKirdy D 1996 *Fundamentals and Applications* ed H Weinstock (Dordrecht: Kluwer–Academic) p 599
[4] Tanaka S 1999 *Japan. J. Appl. Phys.* **38** 505–7
[5] Tanaka S 2000 *IEICE Trans.* **E83-C** 44–8
[6] Tanaka S 2001 *IEEE Trans. Appl. Supercond.* **11** 665–8
[7] Tanaka S 2002 *IEICE Trans. Electron.* **E85-C** 687–90
[8] Tanaka S 2002 *Supercond. Sci. Technol.* **15** 146–9
[9] Huang H, Ding J and McCormick P G 1996 *Mater. Sci. Eng.* A **216** 178–84