Detection System for Metallic Contaminants by Eight-Channel SQUIDs

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Abstract. We developed a magnetic contaminant detection system for industrial products such as electrode foils of lithium ion battery employing eight high-Tc SQUID gradiometers. The system was based on pre-magnetization of a contaminant in an object under test by means of permanent magnets of 0.5 T, which magnetization direction was horizontal, in order to suppress the edge effect from the object composed of magnetic material. The object was conveyed to pass under the eight-channel gradiometer array, in which a pair of four gradiometers was aligned in two rows to cover target foils of several tens mm in width. The magnetization from the contaminant in the object was detected by the gradiometers of averaged flux white noise level of 25 µFlux/Hz^1/2. In case that an iron ball passed just under one gradiometer, an iron ball of about φ30 µm in diameter was successfully detected with a signal to noise ratio (S/N) of 5. From measurement results using an iron ball of about 100 µm in diameter, it was demonstrated that the system had a detectable range of 70 mm in width. These results suggest that the system is a promising tool for the quality control of lithium ion batteries.

1. Introduction
Recently, in the fields of leading-edge industry and the food processing, fine metallic contaminants mixed in the products cause the serious problems. Therefore, the metallic contaminant detection process becomes indispensable on the production lines. In such detection on the lines, precise detection technology is demanded. As the conventional methods, induction coil or x-ray computed tomography scanner have been employed. However, the induction coils are lacking in the sensitivity, and the x-ray computed tomography scanners are lacking in the time resolution. Thus, it is difficult by them to detect the metallic contaminants of less than 100 µm in diameter on the product lines [1-4]. However, there is an urgent need for precision industry products such as lithium-ion electrodes, in which 10µm–order size magnetic contaminants cause some faults. Furthermore, the matrix of industrial products can be magnetized and magnetic signals from the matrix are sometimes sufficiently
large to mask signals from contaminants in many cases. Thus, we have developed a detection system employing high-$T_c$ SQUID gradiometers and horizontal magnetization of such magnetic products in a longitudinal direction [5-7]. Since this system was the prototype which used a few SQUIDs, the detectable area was restricted. In order to detect the electrode with a width of several tens mm on the production line, it is necessary to arrange more sensors to cover the full width of the electrode. In this work, we developed a multi-channel contaminant detection system, in which eight SQUIDs are arranged two-dimensionally. The system characteristics were evaluated.

2. Detection system for metallic contaminants

2.1. Principle of detection of contaminant

A principle of the detection of the contaminant is shown in Figure 1. The contaminant detection method using SQUIDs require to magnetize a specimen with a permanent magnet to detect remnant magnetization of the contaminant. Since lithium ion electrodes often contain magnetic materials, they can be also magnetized. In order to distinguish the magnetization of the specimen and that of the contaminant, we employed the horizontal magnetization method in this work. By using this method, the magnetization field from the specimen appears only from the both ends of it as shown in Figures 1. On the other hand, the contaminant generates a local magnetization field near by the contaminant. By detecting the local magnetic field by a SQUID gradiometer, the signals from the matrix and that from the central contaminant are separable as shown in Figure 2.

Figure 1. Diagram of detection method for fine metallic contaminant.

Figure 2. Signal from metallic contaminant in magnetic matrix measured by high-$T_c$ SQUID gradiometer.
2.2. Detection system

Figure 3 shows the multi-channel high-\( T_c \) SQUID detection system for metallic contaminants, which we have developed. In the system, microscopic cryostat, in which Eight-channel SQUID array was mounted, is installed in the double-layer magnetic shield for reduction of environmental magnetic noise. In addition to the cryostat, the system is composed of flux locked loop (FLL) circuit [8] for eight channels, and a belt conveyor for movement of a specimen and a PC to store data. The dimensions (length \( \times \) width \( \times \) height) of the entire system are 2000 mm \( \times \) 750 mm \( \times \) 1550 mm. This contaminant detection system is aimed to be used in manufacturing process of lithium ion electrodes and the likes, thus the belt conveyor conveys a sample continuously at the speed of about 100 mm/sec.

Eight high-\( T_c \) SQUID gradiometers were mounted at the tips of the sapphire rods using silver paint as adhesive. The rods are connected to a copper tank in the cryostat. As shown in Figure 4, the SQUIDs were arranged with intervals of 28 mm in the lateral direction and 18 mm in the longitudinal direction between the centres of the gradiometers for inspection of a full width of the lithium-ion electrode foil of about 70 mm. By arranging the SQUIDs in two rows, the second series of the 4 SQUIDs can cover the area between the front series of the 4 SQUIDs. Liquid nitrogen is poured in the tank in the cryostat and the SQUID gradiometers are cooled at near 77 K through the sapphire rods. The averaged white noise level of all the SQUID gradiometers was 25 \( \mu \text{V}/\text{Hz}^{1/2} \) at 100 Hz. The magnetized specimen passes under the eight SQUIDs, and the remnant magnetization from the contaminant is measured. We record the outputs of the SQUIDs with a low pass filter with the cut-off frequency of 20 Hz and high pass filter with the cut-off frequency of 0.2 Hz.
3. Characterization of system

3.1. Detection of contaminant on magnetic material

An electrode foil of a lithium-ion battery that we target is often made of magnetic materials. Therefore, we prepared a brass plate specimen as a magnetic material, on which an iron ball of 75 \(\mu\text{m}\) in diameter was fixed. The size of the brass plate used as a magnetic matrix is 100 mm x 10 mm x 1 mm. The brass specimen was inspected by the SQUID system. The sample was magnetized horizontally by the permanent magnet of about 0.5 T, and it was transported under the SQUID array by the belt conveyor. The remnant magnetization from the edges of the brass specimen was measured at 7.35 s and 8.37 s when each edge of the specimen passed under the SQUID, which positioned nearest to the specimen, as shown in Figure 5. On the other hand, a larger signal was detected at 7.85 s when the contaminant passed under the SQUID. From the result, it was demonstrated that it is possible to discriminate the contaminant signal from the matrix signal by this system.

![Figure 5. Detection result of a contaminant on magnetic material.](image)

3.2. Relationship between diameter and signal of contaminant

Magnetic signals from contaminants with different diameters were measured with the developed contaminant detection system. Fine iron (S50C) balls with diameters of 27-95 \(\mu\text{m}\) were prepared as contaminants. Each iron ball was set on a glass plate. The iron balls on the glass plates were conveyed to pass just under one SQUID gradiometer of the array. The lift-off distance between the contaminant and the SQUID array was about 2-2.5 mm. The results are shown in Figure 6. In this experiment, one of the eight SQUID gradiometers was used. The relationship between measured signal amplitude and diameter of contaminant is shown in Figure 7. As shown in the figure, signal amplitude of contaminant was approximately proportional to the square of diameter of contaminant. The averaged peak-to-peak noise in the measurements was about 1.5 m\(\phi_0\). Actually, a signal to noise ratio (S/N) necessary for proper detection of contaminant is 3 or more. From the result in Figure 7, it is suggested that the system can detect an iron ball of 20 \(\mu\text{m}\) in diameter with the S/N of 3. In this study, when an iron ball was in the center of a brass plate, the signal was successfully separated from the magnetization signals of the edges of the plate as shown in Figure 5. The averaged noise level except for the signal of the edges of the plate was 1.5 m\(\phi_0\). From the result in Figure 7, since the signal strength from an iron ball of about 30 \(\mu\text{m}\) in diameter was 10 m\(\phi_0\), it was shown that S/N was more than 3. Under these conditions, it is considered that measurement of \(\phi\) 20 \(\mu\text{m}\) of an iron ball on a brass plate is possible. We used brass plates as an object. However, we are planning to use electrode foil of lithium ion battery in stead of brass plates from now on.

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3.3. Detectable area of system

In order to evaluate the sensitive width of the eight gradiometers, we prepared iron (S50C) balls with a diameter of 95 µm. The lift-off distance between the iron piece and the SQUID gradiometer was 2 mm. The position of the ball placed on the conveyor was changed in steps of 1 mm along the direction perpendicular to the conveyor motion until the entire range of (-38 mm to 46 mm) was covered. The object was magnetized in the same manner and the remnant signal from the iron ball was simultaneously detected by the eight SQUID gradiometers. The peak-to-peak values from Ch.1 and Ch.8 are plotted as a function of the offset distance in Figure 8. The position of each peak is where each sensor is located. The detectable width range of one SQUID gradiometer is estimated to be about 15 mm from Figure 8. By adding all the signals from the eight gradiometers, the valleys between the adjacent gradiometers in the signal outputs can be compensated as shown in Figure 8 by a dotted line. The noise level obtained from time traces was approximately 2 mΦ0. If the detection width was defined as a range where the signal-to-noise ratio (S/N) was greater than 3 (corresponding to a signal level of 6 mΦ0), the detection width of eight SQUID gradiometers was 72 mm.
4. Conclusion

We developed the multi-channel contaminant detection system using eight channel high-$T_{c}$ SQUID gradiometer array and the horizontal magnetization method for the industrial products such as the lithium ion electrodes. It was shown that this system has a capability to detect an iron ball of about 20 µm in diameter with the S/N of 3 or more at a lift-off of about 2 mm. It was demonstrated that the detectable range on the belt conveyer was about 72 mm in the case of the iron ball of 95 µm in diameter, thanks to the two-dimensionally arranged SQUID array. The iron ball with a diameter of 75 µm on the brass plate was detected with enough S/N while separating the signal from the ball from the signals from the plate by use of the gradiometers and the horizontal magnetization. The success of the eight-channel system proposed in this paper encourages us to apply this system to a production line on site.

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