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High T_c SQUID sensor system for non-destructive evaluation

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Abstract

Magnetic field mapping measurements were performed using commercial high-T_c superconducting quantum interference device (SQUID) sensors. The sensors are incorporated in a fully computerized scanning system and were found to be operative only when encased inside a double-layer mu-metal shield. This allowed us to measure signals of the order of 0.5 nT. In all cases, the field map obtained was consistent with the theoretically expected images. In measurements with two samples, the relative orientation and center-to-center separation of the samples could be easily deduced from the field map. Surface flaw detection of unglazed ceramic tiles under an externally applied magnetic field is also discussed. © 2000 Elsevier Science S.A. All rights reserved.

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1. Introduction

The use of low critical temperature (T_c) superconducting quantum interference device (SQUID) sensors in non-destructive evaluation (NDE) was introduced some 10 years ago with the pioneering work of Weinstock and Nisenoff [1,2] as another form of magnetic anomaly detection and demonstrated the detection of flaws in steel pipes and plates. Donaldson's University of Strathclyde group used in 1982 SQUID magnetic gradiometry to detect surface breaking cracks in ferromagnetic steel plates [3]. In the last years, Wikswo's University of Vanderbilt group has applied SQUID magnetometry and gradiometry to a variety of problems in biomagnetism and NDE [4a,4b,5].

A simple description of a SQUID is that of a black box that converts magnetic flux into voltage with ultra-high sensitivity. This allows them to work for a given field sensitivity with much smaller pickup coils than other sensors. In addition to their very high magnetic flux sensitivity, high linearity, wide bandwidth (from DC to 10 kHz) and broad dynamic range (130 dB) renders them suitable for a variety of applications (biomagnetism and geophysics) and, of course, NDE applications. Their ability to operate down to zero frequency allows them to detect deeper flaws (e.g. originating from rivet holes) than traditional eddy current sensors, while their broad dynamic range preserves their high sensitivity in strong DC or noise fields.

For high-T_c SQUIDs, the tremendous improvement in sensor and coil fabrication allowed their use in real applications. The operation in real world environments has been addressed in a number of published reports [6–8] and gradiometer use resulted in system noise levels of 180 $fT/Hz^{1/2}$ at 77 K in unshielded laboratory environments [9]. For optimum performance, SQUID applications in unshielded environments require in general an axial gradiometer with large baseline and a high balance. Electronic gradiometers based on several magnetometers are commonly used. A SQUID vector reference consisting of three magnetometers oriented in the x, y and z directions and with the sensing SQUID being z-oriented was recently implemented for operation in unshielded environment [10] with satisfactory results.

The NDE SQUID magnetometry has been applied to flaw characterization, analysis of magnetic properties of materials and corrosion study. The SQUID measures quite sensitively the magnetic flux at multiple locations in the vicinity of the test object, giving a map or image of the magnetic field. This enables the user to detect, locate and evaluate discontinuities, defects or other imperfections. The magnetic field sources can be intrinsic in the test object (corrosion, magnetized samples) or can be provided

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by an external source (injected current or applied magnetic field).

Wikswo [11] has provided a market oriented overview of SQUIDs for biomagnetism and NDE. High-T_c SQUIDs can be used in practical applications because of the less stringent cooling requirements compared to low-T_c SQUIDs, a factor that allows portability of the system. In addition, liquid nitrogen is considerably cheaper, more readily available and easier to transport than liquid helium. For these reasons, there is considerable effort in establishing the use of high-T_c SQUIDs in a variety of NDE applications [12–17]. Although NDE is necessary in many fields, the two most interesting are aging aircraft and aging and crack development in chemical and nuclear reactor pressure vessels [18].

Our aim in this work is to make an initial assessment of our commercial high-T_c sensors capabilities in zero applied magnetic field. Surface flaw detection of ceramic materials under an externally applied magnetic field is also discussed.

2. Experimental setup and measurement description

2.1. Experimental setup

The system comprises three high-T_c SQUID sensors (offset grain boundary type, Conductus, with a pick-up coil area of 3×3 mm^2) placed at the corners of an equilateral triangle with a side of 21.5 mm. In this way, two electronic planar gradiometers were formed. The SQUID sensors are hermetically sealed in a button-shaped, glass-epoxy (G-10) package containing an integral heater that can heat the SQUIDs in case of flux trapping. All three SQUIDs are placed on the same probe that is housed in a fiberglass liquid nitrogen dewar. The sensor-to-sample distance is variable and can be as small as 5–6 mm. However, in this case, the liquid nitrogen consumption is dramatically increased. The probe and the dewar were bought from Conductus.

The SQUIDs operate in a flux modulation technique (flux-locked loop) as null detectors of magnetic flux. Their field-to-flux coefficients are 8.5, 8.5 and 8.3 nT/Φ_0, respectively, while the field noise was tabulated to be 89, 71 and 216 fT/$\sqrt{\text{Hz}}$, respectively. The SQUID controller also allowed reduction of the low frequency noise by injecting a DC offset current into the modulation coil and thus nulling the DC output of the sensor.

The dewar with the SQUID sensors is surrounded by a cylindrical double-layer mu-metal shield (Kepston Q-Fab) with a layer thickness of 1 mm and an air gap of 2 cm between the two layers. Without the mu-metal shield severe noise problems were encountered and the SQUIDs were unable to operate.

A low-magnetic field *xy* scanning stage (Parker motors), with a 26-μm resolution, permits the displacement of the samples in two orthogonal directions. The stage is placed outside the mu-metal shield to avoid any background signals, even though its remanent field is 1/50th that of the earth's field at a distance of 17 mm above the stage. Mechanical stability of the SQUID sensors is extremely important, especially the isolation of low frequency vibrations, when operating the readout electronics on the most sensitive range [19]. To avoid such problems, the whole system was placed on a two-layer marble table with isolating rubber spacers between the layers.

2.2. Measurement description

In a typical NDE experiment, in zero applied magnetic field, the sample is moved underneath the SQUID sensors by means of a long plexiglass translation arm, penetrating the shield from an opening at one side. The other end of the plexiglass arm was firmly mounted on the scanning stage. A low-friction sample platform support, within a side extension of the shield, was used to avoid any undesirable vibrations caused by the movement of the plexiglass arm. The sample can be moved horizontally 300 mm $\times$ 300 mm (usually a 135 mm $\times$ 135 mm mesh was covered with a 3-mm step in both directions) by a pair of stepper motors (S-6, Parker motors), computer controlled by an AT 6400 card using the included factory software. The SQUIDs were heated and tuned before every scan in order to achieve optimum performance. The tune parameters were always close to the calibration values. The SQUIDs outputs are sampled in each step, allowing a time interval of 250 ms for the SQUIDs to settle before each measurement. The scanning direction was always along the *y*-axis.

We performed measurements of the B_z component of the magnetic field of magnetized samples with the moment horizontally (x–y) and perpendicularly (z) oriented, in order to assess the performance of the sensors in zero applied magnetic field. The first type of sample were pieces of a common magnetic card with dimensions of 3×4 mm^2 (sample I) and 3×5 mm^2 (sample II). The magnetic field on the surface of the card pieces was around 30 G. The second type was a pin 5-mm long and with a diameter of 1 mm, magnetized along its axis (sample III). The magnetic field on the tip of the pin was 10 G.

3. Results

Initially, we measured the field produced by the magnetic card pieces with the moment along the *x*- and *y*-axis. The two orientations of the moment were found to be indistinguishable, except of course from the fact that the observed dipole field was along the *x*- and *y*-axis, respectively.

To estimate the field sensitivity of the sensors we performed a scan of the pin sample. Fig. 1 shows the

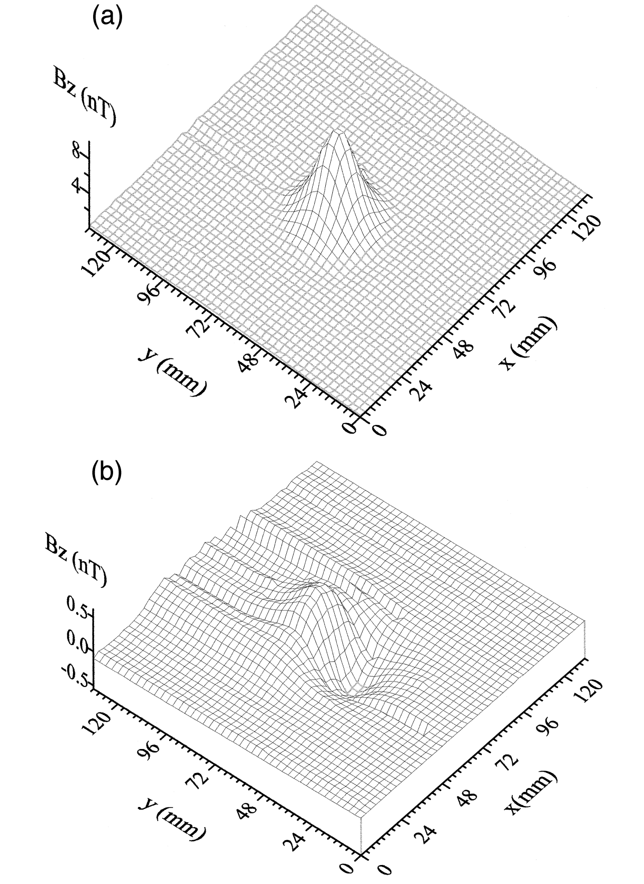

Fig. 1. Surface plot representation of the B_z component of the magnetic field for the pin sample (sample III) in both vertical (upper panel) and horizontal orientations (lower panel). The scale is in nT.

magnetic field map for the pin sample (sample III) placed in vertical (the moment parallel to the z-axis, with a sensor-to-sample distance of 10 mm) and horizontal orientations. For the vertical case, it can be readily seen from the field map that the B_z component of the magnetic field presents axial symmetry as expected and the maximum magnetic field observed is around 10 nT. For the horizontal orientation and with the sample 15 mm away from the sensors, the expected dipole field picture is obtained. The maximum measured field for the horizontal case was around 0.5 nT and the noise in the data is quite small. Some signal artefacts along the scanning direction due to the plexiglass arm are visible in this and in other measurements. These are clearly visible and to some extent degrade the field map in Fig. 1a and b, but nevertheless, the main features are clearly discernible.

Next, the magnet samples I and II were placed in different relative orientations and distances, to determine the system's spatial resolution as a function of the extent and geometrical relationship of the individual magnet-samples. Fig. 2a and b show the magnetic field map (B_z) when the two samples (I and II) are placed 25 mm apart, center-to-center distance, with the sensor-to-sample dis-

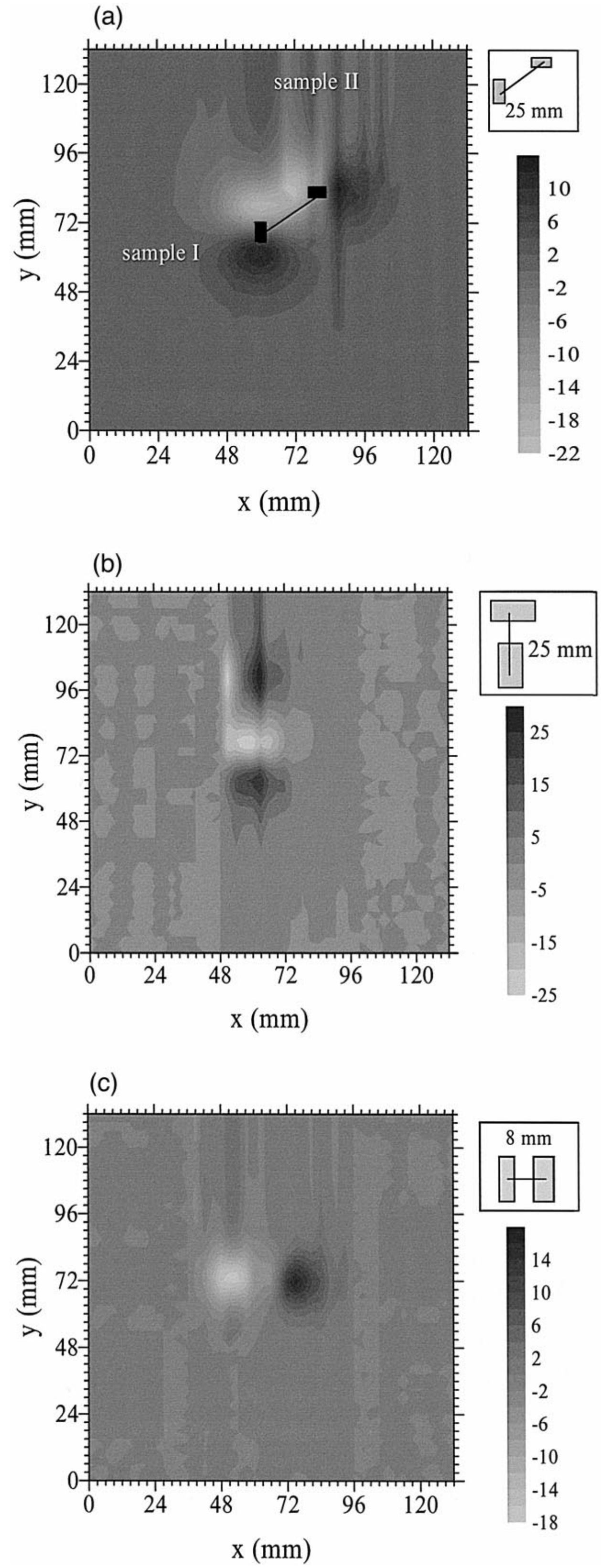

Fig. 2. Isofield maps of the B_z component of the magnetic field when the two samples (I and II) are placed as shown in the drawing at the upper left corner. The scale is in nT.

tances 12 and 10 mm, respectively. For each case, the overall field is the superposition of the magnetic fields produced by the individual samples, considered as dipoles. In the first case (Fig. 2a), the samples axes are orthogonal and this relative orientation is clearly seen from the field map. The center-to-center distance between the two samples can also be deduced accurately from the field map even though the sensor-to-sample distance is almost double the minimum attainable. In the second case (Fig. 2b), the center of sample II lies along the axis of sample I. The relative orientation and center-to-center separation again can be deduced from the field map. Therefore, the spatial resolution even for relatively high sensor-to-sample distance is acceptable. In general, the spatial resolution of a mapping method that uses pickup coils of diameter d and a stand-off distance L is approximately equal to the larger d or L. In our case, the best spatial resolution is determined by the minimum stand-off distance and should be around 5–6 mm. The dimensions of the samples are difficult to determine from the data presented in Fig. 2, mainly because the relatively high stand-off distance, combined with the 3-mm scanning step, smears out their apparent size.

Fig. 2c shows the magnetic field map (B_z) when the two samples (I and II) are placed only 8 mm apart, with the sensor-to-sample distance being 8 mm. The field map obtained resembles a large single dipole. This arises from the strong interaction between the two magnets due to the small sample separation.

Finally, we have also performed scans with an external magnetic field of 5 Oe at the sample, using a system of compensating coils to null the field at the SQUIDs. The coils were powered by a gel cell battery to ensure the stability of the current. The test samples were unglazed ceramic tiles with razor blade scratches 5 mm long, decorated with a paste of superparamagnetic particles (40% MAG/DVB, Seradyn). However, these initial tests were not successful as we observed a loss of lock for the SQUIDs. This is attributed to the small residual field (less than 1 Oe) at the SQUIDs that degraded their performance substantially. Work is underway to improve the compensating coils system.

4. Discussion and conclusions

In conclusion, we have tested an NDE system based on commercial high-T_c SQUID sensors and determined its sensitivity and spatial resolution in zero applied magnetic field inside a magnetic shield. We could measure signals of the order of 0.5 nT. The resolution of the system was adequate to give accurate field maps when the samples are separated by a distance of 25 mm. For smaller separations, the interactions between the magnet samples predominate in the determination of the field map and the resolution of the system is adequate for measuring the resultant field distribution. Initial tests with ceramic samples under an externally applied magnetic field demonstrated the inadequacy of the SQUIDs in even small residual fields. Either better compensating circuitry and coil design or better SQUIDs are necessary. Improved SQUID designs to overcome this problem have been suggested [20].

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Biographies

George Kallias received his BSc in Physics in 1987 and his PhD degree from the University of Athens in 1993. His thesis work involved the experimental study of Hall effect in high-T_c superconducting oxides. At present, he is employed as a post-doctoral fellow by the Institute of Materials Science, NCSR “DEMOKRITOS,” and his recent experimental work is related to the development and evaluation of a SQUID sensor that is based in high-T_c materials.

Eamonn Devlin received his BSc in Physics in 1982 from Dublin University (TCD) and his PhD degree from the University of Birmingham in 1990. His thesis work involved study of transition metal semiconductor compounds. At present, he is employed as a post-doctoral fellow by the Institute of Materials Science, NCSR “DEMOKRITOS,” and his recent experimental work is related to hard magnetic materials and colossal magnetoresistance compounds.

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Dimitrios Niarchos is currently the Director in the Institute of Materials Science and is vice president in the directors board of the NCSR “DEMOKRITOS,” Greece. He received his BSc in Physics in 1972 and his PhD degree from the University of Athens in 1978. Between 1978 and 1981, he was a distinguished post-doctoral fellow at the Argonne National Laboratory, and from 1981 to 1985, he was an Assistant Professor at the Illinois Institute of Technology, Chicago, USA. Since 1985, he is a research scientist at NCSR “DEMOKRITOS,” Greece. His main research areas are in magnetic materials, high-T_c superconductors and sensors based on these materials.