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Superconducting Quantum Interference Devices Based on $YBa_2Cu_3O_{7-x}$ Films for Nondestructive Testing

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Abstract—Original superconducting quantum interference devices (SQUIDs) with a working temperature of 77 K based on high-temperature superconducting (HTSC) $YBa_2Cu_3O_{7-x}$ films can be used for the measurement systems of nondestructive testing using magnetic and eddy-current methods. A dynamic range of 120 dB with respect to the amplitude of the measured signal and a spatial resolution of about 10 μm are reached for the measurement system with the HTSC SQUID in which a receiving loop with a size of 50 μm is placed at a distance of about 0.3 mm from the room-temperature object under study. The sensitivity with respect to magnetic field (4 fT/$\sqrt{\text{Hz}}$ at a temperature of 77 K) of the HTSC SQUID magnetometer with multilayer superconducting flux transformer is sufficient for applications in biomagnetic measurements in magnetocardiography and magnetoencephalography. HTSC SQUID gradiometers with multilayer superconducting flux transformers exhibit stable operation in magnetically unshielded space at a sensitivity of 15 fT/cm $\sqrt{\text{Hz}}$ with respect to the gradient of magnetic field at 77 K. Such a sensitivity is sufficient for the detection of single magnetic particles with a size of about 10 μm at a distance of about 15 mm.

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INTRODUCTION

Methods for nondestructive testing (NDT) are used to verify reliability of the main working properties and parameters of object or single elements (units) in operating systems without their disassembling. The purpose of the NDT is the detection and determination of the location of defects in the object under study and the measurement of the corresponding parameters. Modern NDT methods are used for detection of anomalous deviations in the structure of biological objects, which makes it possible to study living organisms and tissues without deterioration of vital activity. One of the gentlest NDT procedures employs magnetic and eddy-current methods, which are based on the measurement of magnetic anomalies related to internal defects or magnetic properties of the object under study. The magnetic NDT involves passive measurements of magnetic signals generated by the object under study, and the eddy-current NDT is based on the excitation of the induction eddy currents (Foucault currents) in a conducting object and the analysis of the generated electromagnetic field. A significant problem of the NDT lies in an increase in the signal-to-noise ratio owing to an increase in the sensitivity of detectors and/or a decrease in noise and spurious signals. Such a problem can be solved with the aid of superconducting quantum interference devices (SQUIDs), which exhibit maximum sensitivity with respect to magnetic field and a relatively wide dynamic range with respect to the amplitude of the measured signal. The SQUID measurement systems can be used for remote measurements of magnetic anomalies in technical materials and corresponding engineering structures. Note practical interest in the testing of multilayer structures, which can hardly be implemented with the aid of conventional NDT procedures using ultrasonic diagnostics.

The SQUID working principle is based on the measurement of output voltage V_{out} that is induced by magnetic flux Φ_{SQUID} in the SQUID loop with a conversion coefficient of $\partial V_{out}/\partial \Phi_{SQUID} > 10^{10}$ V/Wb that establishes relationship of the input magnetic flux in the SQUID and the output voltage on the signal characteristic $V_{out}(\Phi_{SQUID})$. Note the following advantages of SQUIDs in the NDT procedures: (i) vector properties of the SQUID magnetometers (measurement of the magnetic-field components), (ii) a relatively wide frequency interval of 0–1 GHz, (iii) maximum sensitivity with respect to magnetic field that allows measurement of several physical quantities that can be transformed into magnetic field, (iv) a relatively high sensitivity to variations in magnetic field that remains unchanged in the presence of relatively strong noise,

(v) a relatively high sensitivity with respect to magnetic flux that is reached even at relatively small sizes of SQUID, and (vi) possibility to make detectors that represent various integrated gradiometers with good suppression of noise from remote sources.

SQUIDs based on high-temperature superconducting (HTSC) films exhibit a relatively high working temperature of 77 K, simple and cheap cooling, and higher energy efficiency in comparison with the SQUIDs based on low-temperature superconducting (LTSC) films working at a temperature of 4.2 K. A higher working temperature makes it possible to use smaller distances between detector and object, which is important for an increase in the signal-to-noise ratio and improvement of the spatial resolution in magnetic microscopy upon detection of magnetic impurities or small dipole sources. The HTSC SQUIDs exhibit sensitivity to magnetic field (about 4 $fT/\sqrt{Hz}$) that is comparable with the sensitivity of the LTSC SQUIDs [1–4]. The latent heat of vaporization of liquid nitrogen (161 kJ/L) is significantly greater than the latent heat of vaporization of liquid helium (2.68 kJ/L). Therefore, the evaporation rate of liquid nitrogen is an order of magnitude greater than the evaporation rate of liquid helium is cryostats with comparable sizes. In addition, the application of the cryogenic liquid and/or cryogenic coolers is simpler and cheaper at a temperature of 77 K than at a temperature of 4.2 K. The application of liquid nitrogen as a cooling agent simplifies maintenance of SQUID systems, so that such system can be employed not only in laboratories but also in industrial and field measurement devices.

The HTSC SQUIDs that has been developed by one of the authors at the Forschungszentrum Jülich GmbH were successfully tested in the NDT systems for monitoring of breakage of reinforcement in concrete structures [5], detection of hidden cracks in the inner layers of aircraft fuselage and wheel rims [6], analysis of integrity of plastic structures reinforced with carbon tissue [7], and noncontact testing of semiconductor structures using a laser SQUID microscope [8, 9]. In this work, we consider the HTSC SQUID magnetometers and gradiometers that are used in the NDT systems, magnetic microscopy, and biomagnetic measurements.

1. AUTONOMOUS HTSC SQUIDs WITH A SIZE OF PICK-UP LOOP OF 50 μm

A loop made of an epitaxial HTSC film with two Josephson junctions is the simplest variant of the magnetic-field sensor based on the HTSC SQUID (see Fig. 1). Such SQUIDs measure magnetic field in the absence of additional transformers of magnetic flux and, hence, can be classified as autonomous. In the first approximation, an effective area of the autonomous SQUID with a circular loop coincides with the geometrical area, which is optimal with respect to sensitivity and spatial resolution. The inner and outer diameters of the SQUID loop are about 50 and 70 μm, respectively. An increase in the outer diameter of the SQUID loop allows a proportional increase in the SQUID sensitivity to magnetic field but leads to worsening of the spatial resolution and a decrease in the dynamic range of the SQUID in magnetic fields. The $YBa_2Cu_3O_{7-x}$ (YBCO) films in the SQUIDs exhibit *c* orientation of crystalline lattice, a critical current density of about 5×10^6 A/cm^2 at a temperature of 77 K, and a temperature of superconducting transition of about 91 K. Most Josephson junctions are fabricated on bicrystalline substrates with a disorientation of 24° in the substrate plane. Such bicrystalline junctions have a width of about 1 μm, a critical current of $I_{cr} \sim 30$ μA, and a characteristic voltage of $I_{cr}R_n \sim$ 400 μV at a temperature of 77 K (R_n is the resistance of the junction in normal state). Recently developed HTSC SQUIDs employ Josephson junctions on a step (see [4] and references therein). Such devices have a higher characteristic voltage of up to 600 μV at a temperature of 77 K and are more practical with respect to price and availability of pristine substrates.

In this work, we study various modifications of dc-current SQUIDs (dc-SQUIDs) that represent superconducting-film loops with two Josephson junctions. Signal characteristic $V_{out}(\Phi_{SQUID})$ and sensitivity that is determined by the noise spectral density with respect to magnetic field $b(f)$ are the main measured characteristics of the dc-SQUIDs. The signal characteristic of a dc-SQUID is periodic with respect to Φ_{SQUID} with a period that is equal to a quantum of magnetic flux $\Phi_0 \approx 2.068 \times 10^{-15}$ T m^2 = 2.068 nT μm^2. It is commonly accepted that the SQUID sensitivity to magnetic field $b(f)$ is represented as the noise spectral density at the output of the measurement system that is recalculated to the input. In this case, quantity $b(f)$ represents a sum of SQUID intrinsic noise $b_{SQUID}(f)$, noise of control electronics $b_{el}(f)$, and surrounding noise $b_{ext}(f)$ in the absence of the measured signal. Quantity $b_{sys}(f) = b_{SQUID}(f) + b_{el}(f)$ is the lower-bound limit for the sensitivity of the measurement system that is determined as $b_{sys}(f) = \sqrt{S_B(f)} = k\sqrt{S_\Phi(f)}$, where $\sqrt{S_B(f)}$ and $\sqrt{S_\Phi(f)}$ are the noise spectral densities of the SQUID with respect to magnetic field and flux with including the noise of control electronics while the conversion coefficient of measured magnetic field B_m into magnetic flux Φ_{SQUID} via SQUID loop $k = \partial B_m/\partial\Phi_{SQUID}$ [nT/Φ_0] clearly shows the measured field that corresponds to a single period of signal characteristic $V_{out}(\Phi_{SQUID})$.

The signal characteristic of the dc-SQUID is linearized using electronic circuit with negative feedback that compensates for the deviation of magnetic flux Φ_{SQUID} through the SQUID loop from the working point on dependence $V_{out}(\Phi_{SQUID})$ at which slope $\partial V_{out}/\partial\Phi_{SQUID}$ reaches maximum. In this case, the output signal of magnetometer is proportional to the feedback current and, hence, variation in the external

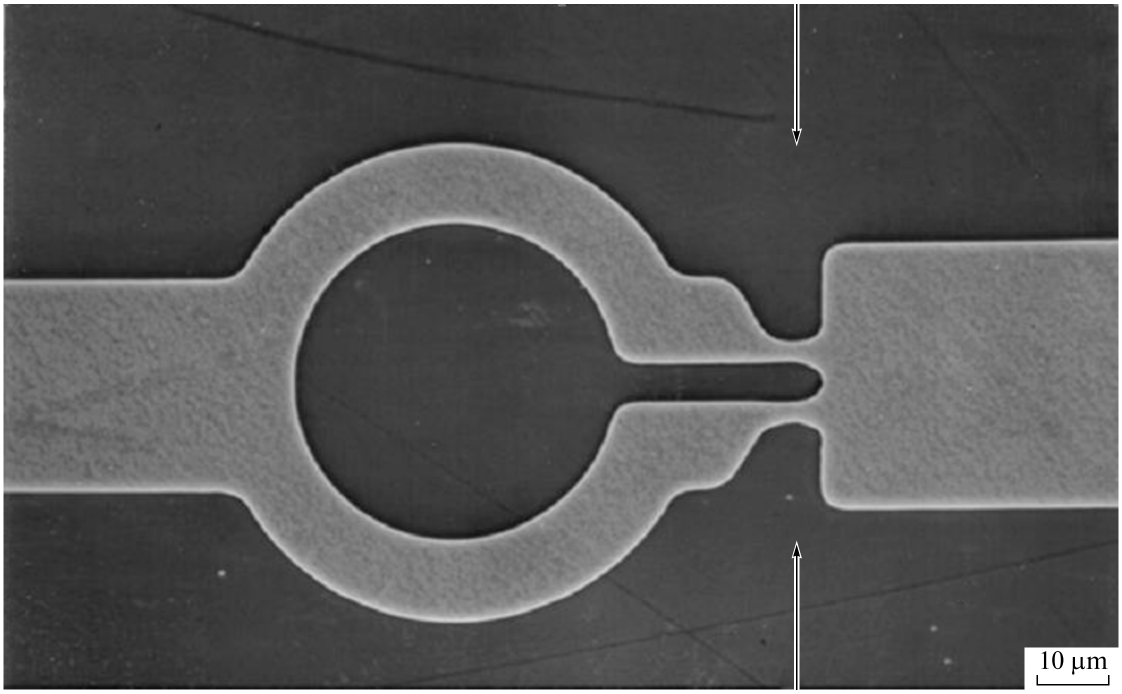

Fig. 1. SEM image of the autonomous HTSC SQUID with bicrystalline junctions (the arrows show the position of the bicrystalline interface in the substrate and HTSC film).

magnetic flux through the SQUID loop. The maximum slope of the signal characteristic is predominantly determined by inductance L_{SQUID} of the SQUID loop and resistance R_{n} according to the expression [10]:

$$\frac{\partial V_{\mathrm{out}}}{\partial \Phi_{\mathrm{SQUID}}} = \frac{4}{\Phi_0}\frac{I_{\mathrm{cr}}R_{\mathrm{n}}}{1+\dfrac{2L_{\mathrm{SQUID}}I_{\mathrm{cr}}}{\Phi_0}}\exp\left(-3.57\pi^2\frac{k_{\mathrm{B}}TL_{\mathrm{SQUID}}}{\Phi_0}\right), \quad (1)$$

where $k_{\mathrm{B}} = 1.38 \times 10^{-23}$ J/K is the Boltzmann constant and T is the absolute temperature.

Conversion coefficient k is calibrated in a three-layer magnetic shield with an internal diameter of about 33 cm. For the autonomous SQUID (Fig. 1), the conversion coefficient of the external magnetic field into SQUID magnetic flux is $k \approx 500$ nT/Φ_0 and modulation amplitude of the signal characteristic ΔV_{out} is about 30 μV. Figure 2 presents noise spectral density with respect to magnetic field $b(f)$ of the measurement system with autonomous SQUID. The measurements were performed in the presence of magnetic shielding and different magnetic fields in the magnetically unshielded space. Most part of the $1/f$ noise of quantity $b(f)$ in the presence of magnetic fields is related to the drift of the power supply of electromagnet and the motion of vortices of magnetic field in the superconducting film of the SQUID body.

The above autonomous SQUIDs were used in [5, 6] and magnetic microscopy [11]. The latter application is related to a unique combination of sensitivity, spatial resolution, and negligible magnetic back action of SQUID on the object under study.

A dynamic range of the magnetometric system of about 120 dB with respect to the amplitude of the measured signal was reached in [12] using the above HTSC SQUIDs. A spatial resolution of about 10 μm was demonstrated in [11] for a distance of about 0.3 mm from the SQUID working at a temperature of 77 K to a room-temperature object under study. Sufficient resolution with respect to magnetic field, relatively high spatial resolution, and possibility of motion in the presence of the Earth magnetic field in the absence of feedback loss in the conventional control electronics of SQUIDs allow NDT applications of such magnetometers. The typical sensitivity of the autonomous HTSC SQUIDs with respect to magnetic field (about 10 pT/$\sqrt{\mathrm{Hz}}$) is sufficient for sounding of aircraft fuselage using eddy currents and monitoring of the integrity of reinforcements of concrete structures using magnetization: a magnetic response of 1 nT can be measured at a signal-to-noise ratio of about 100 at an amplitude of sounding signal of about 10 mT.

2. HTSC SQUID MAGNETOMETERS WITH SUPERCONDUCTING FLUX TRANSFORMER

The best sensitivity of the SQUID magnetometers with respect to magnetic field is reached using a superconducting transformer of magnetic flux with a rela-

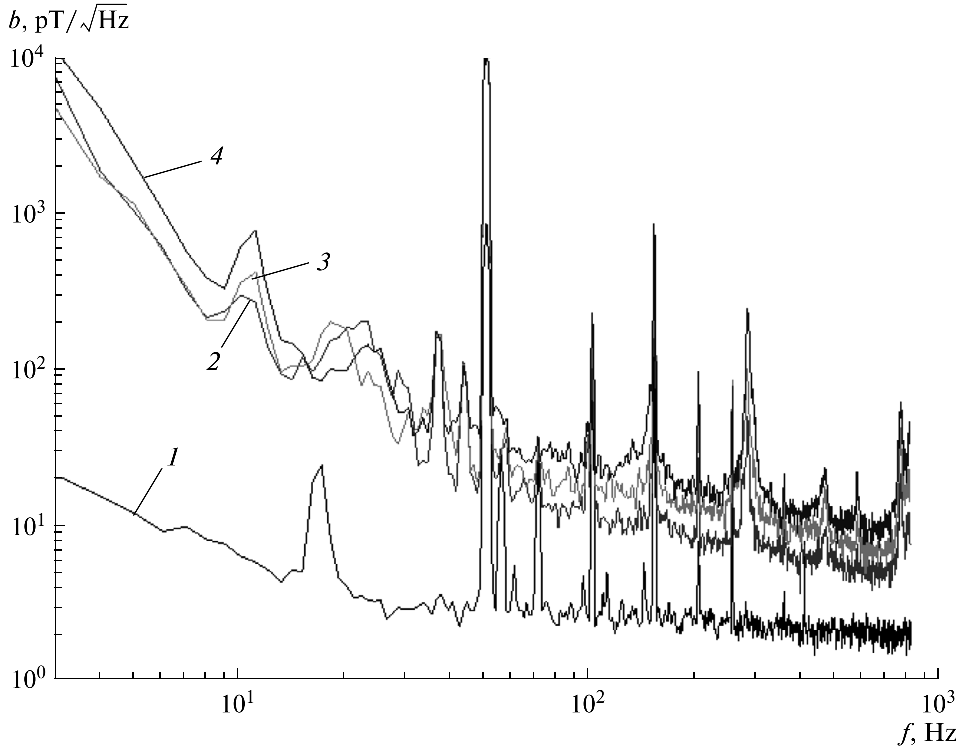

Fig. 2. Noise spectral density $b(f)$ of an autonomous HTSC SQUID in the presence of magnetic fields B = (*1*) 0, (*2*) 4, (*3*) 8, and (*4*) 13 mT that are generated by an electromagnet with iron core and power supply with current stabilization.

tively large area of the receiving loop and multiturn input coil that provides the maximum transfer of magnetic flux to the SQUID loop. The most practical configuration of the HTSC SQUID magnetometer employs the flip-chip connection of the SQUID and flux transformer that are fabricated on different substrates. For the pick-up loop of the flux transformer with a size of 16 mm × 16 mm, the best resolution with respect to magnetic field of the flip-chip magnetometers is about 4 fT/$\sqrt{\text{Hz}}$ at a temperature of 77 K and a conversion coefficient of $k = 0.45$ nT/Φ_0 [3, 4]. The last modification of such a HTSC SQUID magnetometer (Fig. 3) contains a flux transformer with a circular (20 mm) loop and the outer diameter of the encapsulated structure is 24 mm [13]. The conversion coefficient of such a system is $k = 0.4$ nT/Φ_0.

The limiting sensitivity with respect to magnetic field of the SQUID measurement system that is inductively coupled with a multiturn input coil of the superconducting flux transformer can be estimated as

$$b = b_{\text{sys}} + b_{\text{ext}} \approx b_{\text{sys}} = \sqrt{S_B} = \frac{L_{\text{r}} + L_{\text{c}}}{\alpha A_{\text{r}} \sqrt{L_{\text{c}} L_{\text{SQUID}}}} \sqrt{S_\Phi}, \tag{2}$$

where A_{r} and L_{r} are the area and inductance of the receiving loop of the flux transformer, respectively; α is the coupling coefficient of the SQUID and input coil of the flux transformer; and L_{c} is the inductance of the input coil of the flux transformer. The flux noise spectral density of the SQUID and control electronics $\sqrt{S_\Phi}$ is predominantly determined by the thermal fluctuations in the Josephson junctions, maximum slope of the signal characteristic, and the noise of preamplifier of control electronics $S_{\text{el}} \approx 0.04$ (nV)^2:

$$S_\Phi = S_V \Big/ \left(\frac{\partial V_{\text{out}}}{\partial \Phi_{\text{SQUID}}}\right)^2 \approx \left\{ \frac{12 k_{\text{B}} T}{R_{\text{n}}} \times \left[\frac{R_{\text{n}}^2}{2} + \frac{L_{\text{SQUID}}^2}{4} \left(\frac{\partial V_{\text{out}}}{\partial \Phi_{\text{SQUID}}}\right)^2 \right] + S_{\text{el}} \right\} \Big/ \left(\frac{\partial V_{\text{out}}}{\partial \Phi_{\text{SQUID}}}\right)^2. \tag{3}$$

The above high-sensitivity sensors of magnetic field can be used in the measurement systems for the NDT of biological objects. The sensors were employed for magnetocardiographic measurements [14] and magnetoencephalography [4, 13, 15]. The HTSC SQUIDs can also be used in scanning systems for measurement of magnetic susceptibility of biological objects aimed at rapid (pre- and intraoperative) detection of malignancies using marking of tumor cells with magnetic nanoparticles as a contrast medium [16]. The SQUIDs can be used for the detection of metal and magnetic impurities at food-industry conveyers [17]. The devices can also be helpful for testing of lithium batteries (detection of iron impurities) and prevention

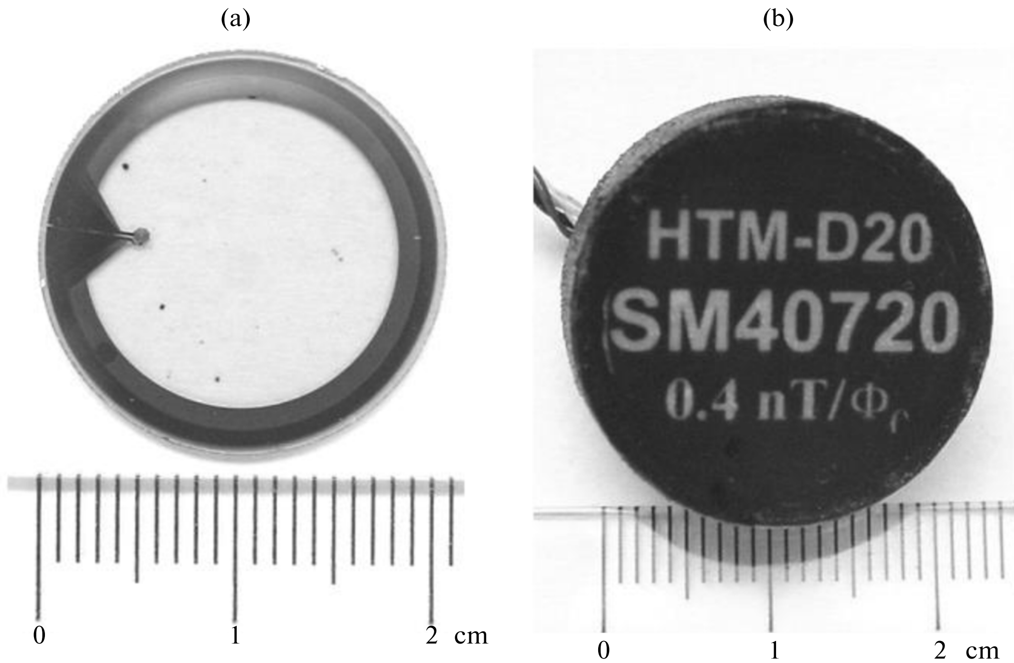

Fig. 3. (a) Multilayer HTSC flux transformer with a 20-mm receiving loop and 14-turn coupling coil and (b) encapsulated SQUID magnetometer that contains such a flux transformer [13].

of the dissociation of the impurities to electrolyte and consequent short-circuiting of batteries.

3. HTSC SQUID GRADIENT METERS WITH SUPERCONDUCTING FLUX TRANSFORMERS

A physical problem related to the practical application of SQUIDs in NDT lies in the separation of relatively weak signals in the presence of strong magnetic noise. High-sensitivity measurements of magnetic signals in magnetically unshielded space are difficult to implement owing to the magnetic interference at mains frequency with harmonics of such a frequency and the presence of various low-frequency noises. The sensitivity of modern HTSC dc-SQUID sensors of magnetic field is better than 10 $\mathrm{fT}/\sqrt{\mathrm{Hz}}$ at a working temperature of 77 K. At the same time, the amplitude of typical magnetic noise is greater than 100 nT under laboratory conditions and the Earth magnetic field is about 50 μT. Spurious signals related to the effect of magnetic field on the SQUID sensor or motion (vibration) of the sensor in the Earth magnetic field may be greater than the analyzed signal by ten orders of magnitude.

The spatial filtering of signal using gradient measurements is the most efficient and widely used method for elimination of noise signals. Normally, magnetic field B of the sources that are located at relatively large distances from the measurement system is more spatially uniform in comparison with the field of a closely located source, so that gradient components $\partial^n B/\partial r^n$ ($n = 1, 2, \ldots$ is the order of gradient meter) are significantly smaller than the gradient components of a closely located source. For example, magnetic field B of a magnetic dipole decreases as $1/r^3$ whereas corresponding gradients $\partial^n B/\partial r^n$ decrease faster as $1/r^{3+n}$. The measurement of the gradient components of the magnetic field makes it possible to detect the magnetic signal of a closely located source and suppress admixing of magnetic noise from distant sources.

In the presence of strong magnetic noise and in the absence of magnetic shielding, we may use a planar HTSC SQUID gradiometer of the first order that has a gradiometric multilayer superconducting flux transformer with a size of about 1 cm (Fig. 4). Such a HTSC SQUID gradiometer was employed in [18, 19] for the detection of magnetic impurities in the construction materials under laboratory conditions in the absence of magnetic shielding.

Uniform magnetic field induces shielding currents along the perimeter of the gradiometer and does not contribute to the current in the input coil. Only nonuniform magnetic field can induce the supercurrent that flows through the input coil located at the diagonal of the flux transformer. The supercurrent that is induced in the input coil is equal to the difference of supercurrents that are induced in the loops of the pickup coil and, hence, proportional to the gradient of external magnetic field.

Different configurations of the pick-up coil of the gradiometer are possible for particular applications. As in the systems with flip-chip magnetometers (see pre-

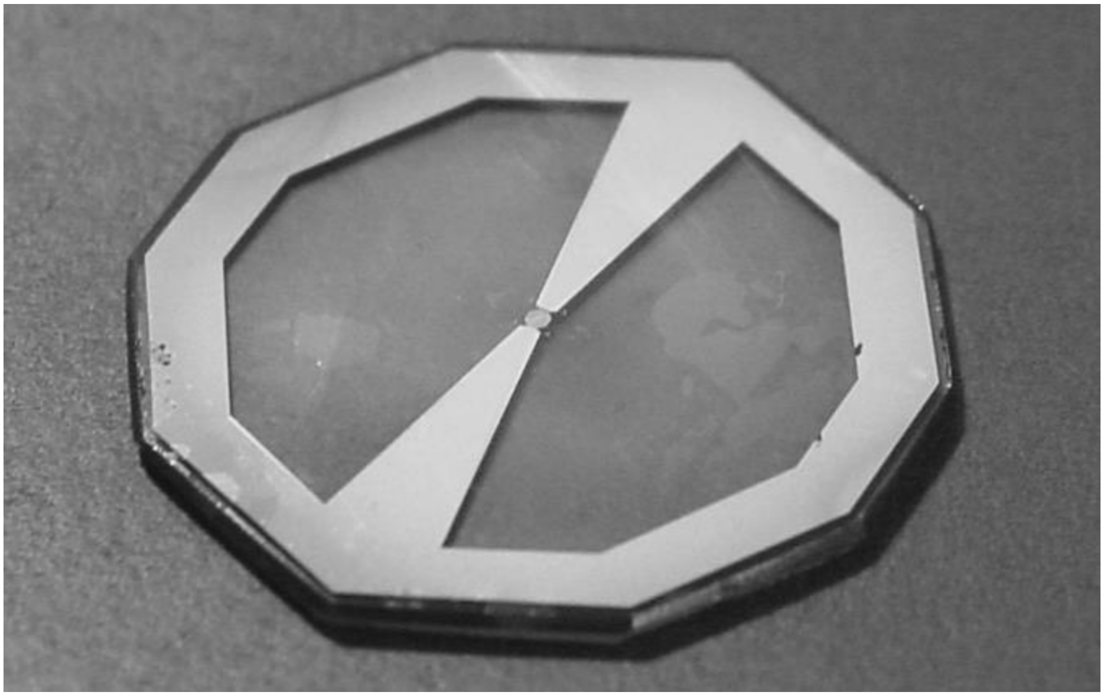

Fig. 4. Photograph of the HTSC SQUID gradient meter with a multilayer flux transformer and a base length of 1 cm.

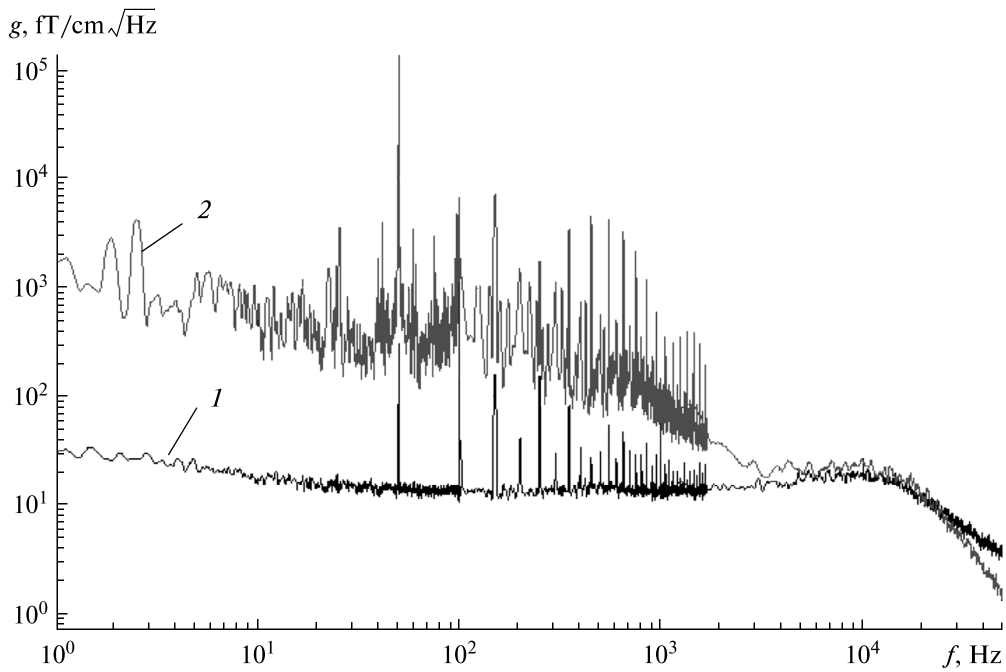

Fig. 5. Noise spectral densities with respect to magnetic-field gradient $g(f)$ of the planar HTSC SQUID gradient meter with a base size of 1 cm that are measured in (*1*) magnetic shield and (*2*) magnetically unshielded space.

vious section), the application of a multilayer multiturn input coil leads to improvement of inductive coupling of the pick-up coil and SQUID in the gradiometer. Figure 5 shows examples of noise spectral density of magnetic field gradient $g(f)$ for such a gradiometer that are measured in a magnetic shield and magnetically unshielded space. Similarly to noise spectral density of magnetometer $b(f)$, quantity $g(f)$ contains the intrinsic noise of the measurement system and external noise.

Sensitivity of gradiometer $(\partial B_z/\partial x)/\Phi_0$ is calibrated outside magnetic shield using two parallel wires located at distance $2d$ from each other. Identical currents I flow in the wires. At altitude h above the wires, the vertical component of magnetic field B_z is given by

$$B_z(x) = -\frac{\mu_0 I}{2}\left(\frac{x+d}{(x+d)^2+h^2}+\frac{x-d}{(x-d)^2+h^2}\right), \quad (4)$$

where $\mu_0 = 4\pi \times 10^{-7}$ H m. At $I = 1$ mA, $h = 3.7$ cm, and $d = 1.6$ cm, field gradient $\partial B_z/\partial x$ is almost constant (1.7 nT/cm) at x ranging from −1 to 1 cm. For the HTSC SQUID gradiometer with the flux transformer (Fig. 4), the measured $(\partial B_z/\partial x)/\Phi_0$ is about 1 nT/(cm Φ_0).

Single magnetic particles with a size of about several tens of micrometers were detected and localized with the aid of the measurement system using the HTSC SQUID gradiometer. Magnetic dipole moments of such particles can also be determined. A quantitative analysis of the scanning data was based on the following formula for magnetic field $\vec{B}$ of a magnetic dipole:

$$\vec{B} = \frac{\mu_0}{4\pi}\left[\frac{3(\vec{P}\cdot\vec{r})\vec{r}}{r^5} - \frac{\vec{P}}{r^3}\right], \quad (5)$$

where $\vec{P}$ [A m^2] is the magnetic moment of the dipole and $\vec{r}$ [m] is the distance from the dipole to the measurement point. The estimations of [20] show that the minimum size of the detected magnetic particles (about 10 μm) is in good agreement with the experimental data.

The sensitivity of the gradiometer can be improved using an increase in the area of receiving coils and the distance between them. Both parameters are limited by the size of substrate and modern technologies of the formation and structuring of multilayer metal–oxide films. At present, multilayer HTSC structures can be fabricated at substrates with a diameter of up to 30 mm. Note that the outer diameter of the optimal structure is no greater than 25 mm, since the quality of the HTSC films at distances of about 1 mm from the substrate edge is lower than the film quality in the central part. In the future, planar HTSC gradiometers with multilayer flux transformers will be fabricated on the MgO single-crystal substrates with a size of up to 100 mm. The application of the planar HTSC gradiometers is necessitated by the absence of sufficiently thin and flexible HTSC wire needed for the fabrication of axial gradiometric flux transformers that are most widely used in the HTSC SQUID gradiometers working in magnetically unshielded space [21, 22].

CONCLUSIONS

We have developed and studied HTSC SQUIDs for several practical applications including biomagnetic diagnostic systems and devices for noncontact testing of semiconductor structures using a laser SQUID microscope. The experimental signal-to-noise ratios are on the same order of magnitude with the signal-to-noise ratios of the systems based on the LTSC SQUIDs. Such results and the existing data for the NDT SQUID systems indicate a possibility of the construction of practical devices for NDT of materials and industrial items at a working temperature of 77 K.

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