Bull. Mater. Sci., Vol. 25, No. 2, April 2002, pp. 101–107. © Indian Academy of Sciences.

Non-destructive evaluation of defects in ferromagnetic plates using a sensitive magnetic sensor based on second harmonic response of superconducting $Bi_{1.6}Pb_{0.4}Sr_2Ca_2Cu_3O_{10+\delta}$ pellet

ABHIJIT RAY and T K DEY*

Cryogenic Engineering Centre, Indian Institute of Technology, Kharagpur 721 302, India

MS received 27 August 2001

Abstract. **The characteristics of a magnetic sensor, based on the non-linear electromagnetic response of the weak links present in polycrystalline BPSCCO superconductor are reported. The second harmonic response of the sensor in an alternating magnetic field at 40 kHz and at 77 K being a strong linear function of low d.c. field is utilized for magnetic field sensing. The noise limited resolution of the sensor is found to be 3.16×10^{-9} T/√Hz for $H_{a.c.} = 16$ G and frequency 40 kHz. The magnetic sensor has been applied for non-destructive detection of various types of flaws in ferromagnetic plates and also for detection of small magnetic inclusions in a non-magnetic matrix. Our results suggest that the 2*f* response based BPSCCO superconductive magnetometer has potential for its application in the area of non-destructive evaluation of defects in ferromagnetic materials.**

Keywords. **BPSCCO superconductor; second harmonic response; magnetic sensor; non-destructive evaluation.**

1. Introduction

Non-linear electromagnetic response observed in polycrystalline ceramic superconductors under low magnetic field is one of the unique features of CuO based superconducting perovskites. These materials being agglomerates of anisotropic grains separated by non stoichiometric interface materials which behave as weak links, the magnetic field can easily penetrate into the inter granular regions and as a result, the low field magnetic behaviour of polycrystalline HTSCs is dictated dominantly by the response of these weak links to the applied field (Muller *et al* 1989; Ishida and Goldfarb 1990; Kim *et al* 1991; Ghatak *et al* 1992). In presence of an a.c. magnetic field of frequency (*f*), the polycrystalline HTSCs generate only the odd harmonics for $T < T_c$. However, superposition of a small d.c. field brings the appearance of even harmonics and amplitudes of the even harmonics are strong linear function of applied d.c. field. Origin of the harmonic generation and its modulation with d.c. field is generally explained in light of various models, viz. magneto resistive model (Xenikos and Lamberger 1990), critical state models (Muller 1990; Qin and Ong 1999) and also by assuming the material as a collection of superconducting loops with weak links (Lam and Jeffries 1989; Navarro *et al* 1990). From application point of view, the observed linear dependence of the amplitude of the second harmonic response with low d.c. magnetic field can be exploited as a highly sensitive magnetic field sensor (Gallop *et al* 1988, 1989; Gielisse *et al* 1991; Khare *et al* 1997; Dey *et al* 1999).

Magnetic sensors, with the capability of sensing very low magnetic fields, are of great technical and commercial significance. One of the potential areas of applications is non-destructive evaluation/detection of defects in electrically conductive magnetic and non-magnetic materials. NDE using any type of magnetometers or gradiometers basically probes the local anomalies in the magnetic or electro magnetic stray fields of the object under test (Braginski and Krause 2000). Such probing consists in the detection of surface or, sub-surface cracks/voids in ferromagnetic objects, as well as, presence of magnetic inclusions in a non-magnetic matrix by sensing the magnetic flux distortion, or, leakage above the surface. Using two superconducting YBCO based magnetic sensors in gradiometer configuration, Buckley *et al* (1991) reported detection of defects in ferromagnetic materials. In the present communication, we report the characteristics of a sensitive magnetic sensor based on second harmonic response of a sintered BPSCCO superconducting pellet having noise sensitivity of $\sim 10^{-9}$ T/$\sqrt{\text{Hz}}$ and demonstrate its possible application in the detection of different types of defects in ferromagnetic plates, including

*Author for correspondence

detection of small magnetic inclusions in a non-magnetic matrix.

2. Experimental

The BPSCCO sensor used in the present work was prepared from high quality $Bi_{1\cdot6}Pb_{0\cdot4}Sr_2Ca_2Cu_3O_{10+\delta}$ precursor powder obtained from M/s CAN Superconductors, Czech Republic. The said powder was cold pressed in the form of cylindrical pellets (dia. ~ 12 mm, thickness ~ 4 mm) with a pressure of 5·5 MPa and was subsequently sintered at 890°C for 6 h in air. The pellet was furnace cooled to room temperature slowly in air. The prepared sample was superconducting with T_{C0} ~ 100 K, J_{C0} = 60 A/cm^2 and had a density of 5·39 g·cm^{-3}. The SEM picture of the prepared sample confirmed the existence of small grains and significant porosity (~ 40%). It may be pointed out that for the present applications, it is advantageous to have sample with lower density (higher porosity) and lower J_C. This is because, the second harmonic amplitude at any particular d.c. field ($H_{d.c.}$), for a constant a.c. field ($H_{a.c.}$) of frequency (f) is inversely proportional to J_C and is given by (Gielisse *et al* 1991):

$$V_{2f} = \omega H_{d.c.} H^3_{a.c.} AN/J_C(H).$$

The schematic of the experimental arrangements used for the characterization of the BPSCCO magnetic sensor is shown in figure 1. The sensor is placed centrally at the centre of a small a.c. driving coil, which generates an a.c. magnetic field and drives the superconductor in and out of the mixed state. A pick up coil (50 turns, 46 swg enameled copper wire) is wound directly on the sample. The components of voltage, corresponding to the second harmonic of the applied frequency induced in the pick up coil, are detected by a lock-in-amplifier (Stanford, model 850). The sensor with the pick up coil and the a.c. solenoid are located coaxially inside a d.c. solenoid, which provides the background d.c. field ($H_{d.c.}$). This background d.c. field produced by the solenoid was precalibrated by a Lakeshore Hall sensor. If the applied sinusoidal a.c. field ($H_{a.c.} \sin \omega t$) is large enough to drive the sample in the mixed state, its magnetic response becomes non-linear. The voltage developed across the detection coil is proportional to the time derivative of the magnetic induction $B(t)$, and can be written as

$$V(t) = NA\omega H_{a.c.} \sum_{n=1,2,3} n[(\mu'_n \sin(n\omega t) - \mu''_n \cos(n\omega t))]$$
$$= \sum_n V_{nf},$$

where, μ'_n and μ''_n are respectively the real and imaginary components of the measured harmonic susceptibilities of the sample, NA the area turns of the secondary coil.

3. Results and discussion

3.1 *BPSCCO magnetic sensor*

Typical $2f$ response of the present BPSCCO magnetic sensor as a function of d.c. field between 0 and 130 G at 30 kHz for different a.c. field amplitudes and also at frequencies between 10 and 30 kHz for $H_{a.c.}$ = 13 G are shown in figures 2a and b, respectively. The second

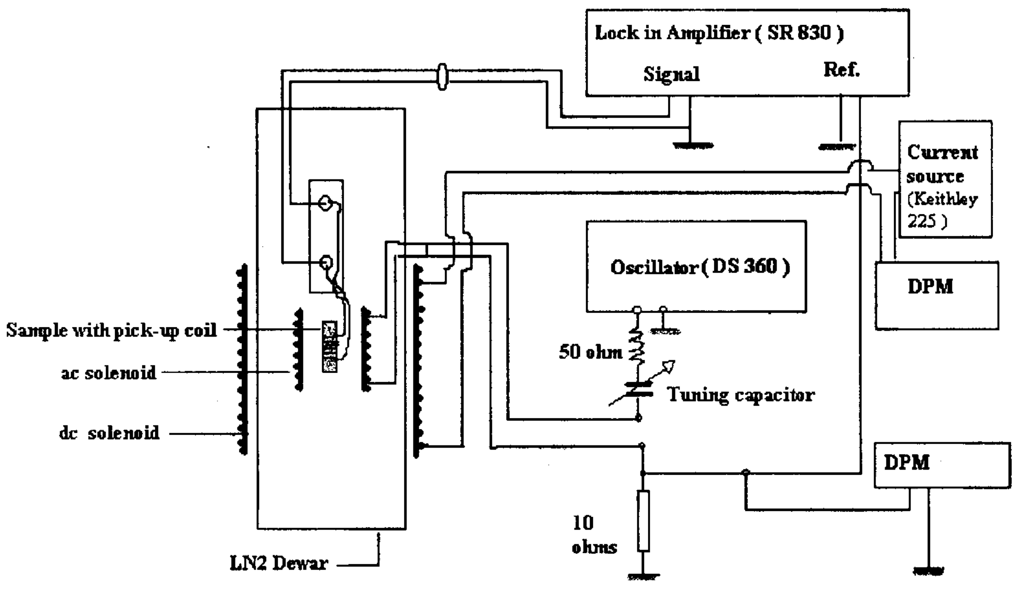

Figure 1. Schematic of the experimental arrangement for the characterization of second harmonic response based BPSCCO magnetic sensor.

harmonic amplitude (V_{2f}) initially increases sharply and linearly at low d.c. field, attains a maximum and then starts decreasing. Eventually it approaches a nearly constant value at higher magnetic fields. It may be noted that the $2f$ response varies sharply and nearly linearly in presence of a small varying d.c. magnetic field. All these features of $2f$ response in ceramic superconductors have been reported earlier by various authors and have been well accounted for by the critical state models (Muller *et al* 1989; Ishida *et al* 1990) and assuming the granular nature of the ceramic superconductors. A systematic study of the variation of V_{2f} with $H_{\text{d.c.}}$ corresponding to different a.c. fields and for frequencies between 10 and 40 kHz was undertaken to characterize the prepared BPSCCO pellet for use as sensor for low magnetic fields. The points in figures 3a–d are the experimental data in the range $0 < H_{\text{d.c.}} < 3$ G and the corresponding solid lines represent the best-fit linear relation. The slopes ($dV_{2f}/dH_{\text{d.c.}}$) of these linear fits are given in table 1. It may be seen from table 1 that the rate of increase in the response is higher for higher a.c. field amplitudes and also for higher driving frequencies. For the present sensor, V_{2f} response with $H_{\text{d.c.}}$ as high as 3·16 mV/G is obtained corresponding to an a.c. field amplitude of 16 G and driving frequency of 40 kHz. The field noise ($\sqrt{S_B}$), or the smallest field change, which could be detected by the sensor, is estimated from

$$\sqrt{S_B} = [\Delta V/(\delta V/\delta H_{\text{d.c.}})],$$

where, ΔV is the voltage noise at the output of the lock in amplifier and ($\delta V/\delta H_{\text{d.c.}}$) the transfer function of the sensor. The value of ($\delta V/\delta H_{\text{d.c.}}$) is known from the slope of the linear region of V_{2f} vs $H_{\text{d.c.}}$ plots. The estimated noise sensitivity ($\sqrt{S_B}$) at different frequencies and $H_{\text{a.c.}}$ for the present sensor is shown in table 1. It may be noted that the increase observed in ($\delta V/\delta H_{\text{d.c.}}$) is related to increase in flux penetration due to increasing $H_{\text{a.c.}}$. In other words, the field noise ($\sqrt{S_B}$) should continue to decrease and finally attains a minimum, once the a.c. field fully penetrates the sample. The highest noise sensitivity ($\sqrt{S_B}$) for our BPSCCO sensor was found to be $3{\cdot}16 \times 10^{-9}$ T/$\sqrt{\text{Hz}}$ corresponding to $H_{\text{a.c.}} = 16$ G and $f = 40$ kHz and is nearly five times higher than that reported for BSCCO thick film magnetic sensor (Khare *et al* 1997). The noise at the output of this type of sensors has contributions from three different sources, viz. the coil, the detection electronics and the random flux motion in the superconducting sensor itself. The theoretical limit of $\sqrt{S_B}$ for this class of sensor was estimated to be $\sim 10^{-10}$ T/$\sqrt{\text{Hz}}$ (Gallop *et al* 1989). Nevertheless, the

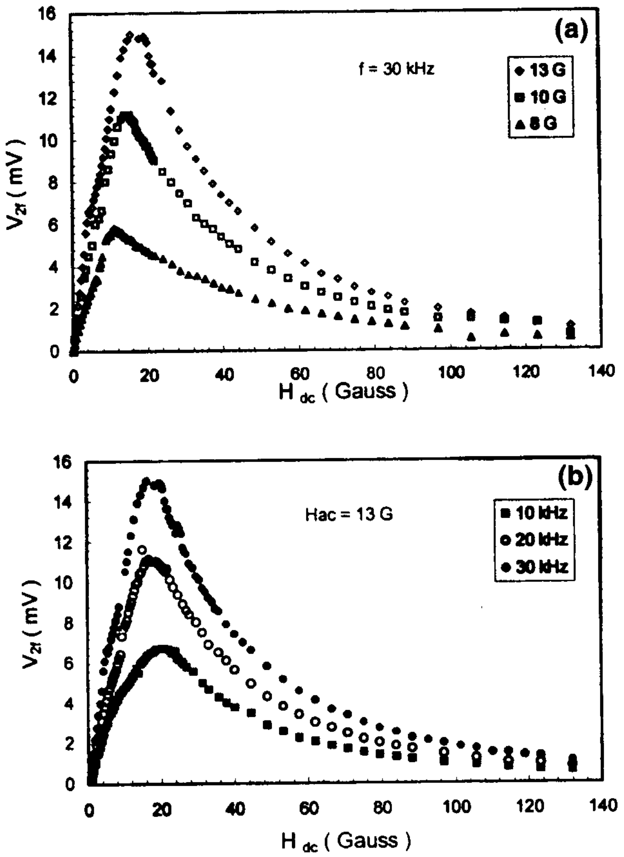

Figure 2. Second harmonic amplitude vs $H_{\text{d.c.}}$ at 77 K (**a**) at various $H_{\text{a.c.}}$ for $f = 30$ kHz and (**b**) at various frequencies for $H_{\text{a.c.}} = 13$ G.

Table 1. Slopes of the best linear fits shown in figures 3a–d and the field noise ($\sqrt{S_B}$) calculated for different a.c. fields ($H_{\text{a.c.}}$) and frequencies (f).

	$H_{\text{a.c.}} = 8$ G		$H_{\text{a.c.}} = 10$ G		$H_{\text{a.c.}} = 13$ G		$H_{\text{a.c.}} = 16$ G	
Frequency (kHz)	$\left(\frac{dV_{2f}}{dH_{\text{d.c.}}}\right)$ (mV/G)	$\sqrt{S_B}$ (T/Hz$^{1/2}$)	$\left(\frac{dV_{2f}}{dH_{\text{d.c.}}}\right)$ (mV/G)	$\sqrt{S_B}$ (T/Hz$^{1/2}$)	$\left(\frac{dV_{2f}}{dH_{\text{d.c.}}}\right)$ (mV/G)	$\sqrt{S_B}$ (T/Hz$^{1/2}$)	$\left(\frac{dV_{2f}}{dH_{\text{d.c.}}}\right)$ (mV/G)	$\sqrt{S_B}$ (T/Hz$^{1/2}$)
10	0·151	$6{\cdot}62 \times 10^{-8}$	0·251	$3{\cdot}98 \times 10^{-8}$	0·402	$2{\cdot}48 \times 10^{-8}$	0·5	$2{\cdot}00 \times 10^{-8}$
20	0·334	$2{\cdot}99 \times 10^{-8}$	0·515	$1{\cdot}94 \times 10^{-8}$	0·817	$1{\cdot}22 \times 10^{-8}$	0·91	$1{\cdot}09 \times 10^{-8}$
30	0·484	$2{\cdot}06 \times 10^{-8}$	0·722	$1{\cdot}38 \times 10^{-8}$	1·143	$8{\cdot}74 \times 10^{-9}$	1·565	$6{\cdot}38 \times 10^{-9}$
40	0·727	$1{\cdot}37 \times 10^{-8}$	1·257	$7{\cdot}95 \times 10^{-9}$	1·920	$5{\cdot}20 \times 10^{-9}$	3·161	$3{\cdot}16 \times 10^{-9}$

present magnetic sensor is not comparable in sensitivity of a high T_C SQUID, however, it has the advantage of moderately high sensitivity, high dynamic range, simplicity of design and fabrication and truly inexpensive.

3.2 *NDE using 2f response of BPSCCO magnetic sensor*

Strong and linear response of the second harmonic amplitude of BPSCCO magnetic sensor at low d.c. fields has strong potential for various applications, one of which is in the area of non-destructive evaluation of defects in ferromagnetic plates. Basically, magnetic effects are explained by the concept of electromagnetic fields, which can be imagined as lines of magnetic force, extending through space. When a ferromagnetic specimen is magnetized, the magnetic lines predominantly remain inside the specimen. However, presence of a crack or, a subsurface flaw in the material produces distortion in the distribution of magnetic field inside the material, and causes local flux leakage/distortion around the defects. The magnitude of the distortion (or flux leakage) is determined by the size and shape of the defects. The present method of non-destructive crack detection in ferromagnetic plate using the BPSCCO magnetic sensor depends on its capability to detect the local magnetic flux distortion.

The schematic diagram of the experimental set-up for the NDE of ferromagnetic plate using the BPSCCO magnetic sensor is shown in figure 4. The sensor ($\phi = 2$ mm, length = 5 mm) with 50 turns of pick-up coil is aligned

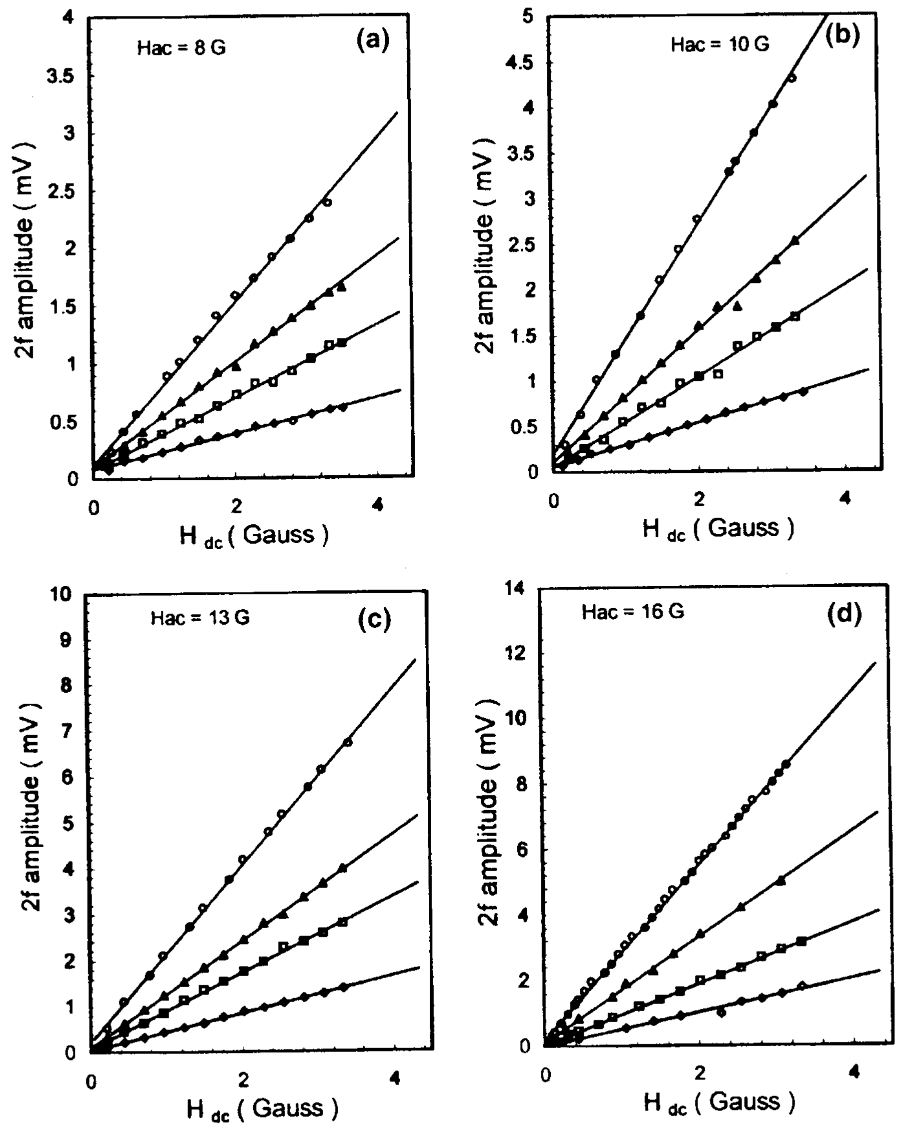

Figure 3. (a)–(d). V_{2f} as a function of d.c. fields at 77 K for different $H_{a.c.}$ and for frequencies 10 kHz (◊), 20 kHz (□), 30 kHz (Δ) and 40 kHz (O). Solid lines are the best fitted linear relations.

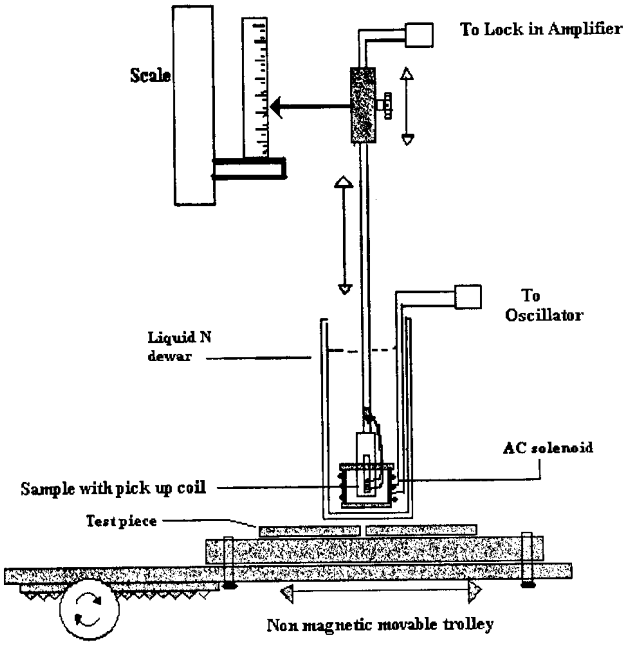

Figure 4. Schematic of the experimental arrangement for NDE of defects in ferromagnetic test pieces using BPSCCO sensor.

axially in the centre of a small drive solenoid (250 turns). The drive solenoid is fed with 40 kHz signal from an Ultra Low Distortion Function Generator (SRS model DS360) and it produces an a.c. field of ~ 10 G. The sample and the a.c. solenoid are kept immersed in a liquid nitrogen glass dewar, mounted appropriately on a non magnetic, non metallic stand. Suitable arrangement has been provided for smooth up and down vertical movement of the sensor along with the a.c. coil assembly, so as to vary the 'stand-off' distance. The specimen under test can be moved underneath the dewar by mounting it on a non-magnetic movable table, provided with vernier scale. The detection electronics is similar to those used for the evaluation of the magnetic response of BPSCCO sensor.

Several types of test pieces were prepared to examine the capability and usefulness of our single sensor based NDE device. The ferromagnetic test pieces are lightly magnetized using a permanent magnet prior to test run. Figures 5a–c show the second harmonic signal for a scan over three steel plates, each containing one slot of width 0·9 mm, 2·5 mm and 5 mm, respectively. Sharp and distinct drop in the sensor output signal is observed as the slotted

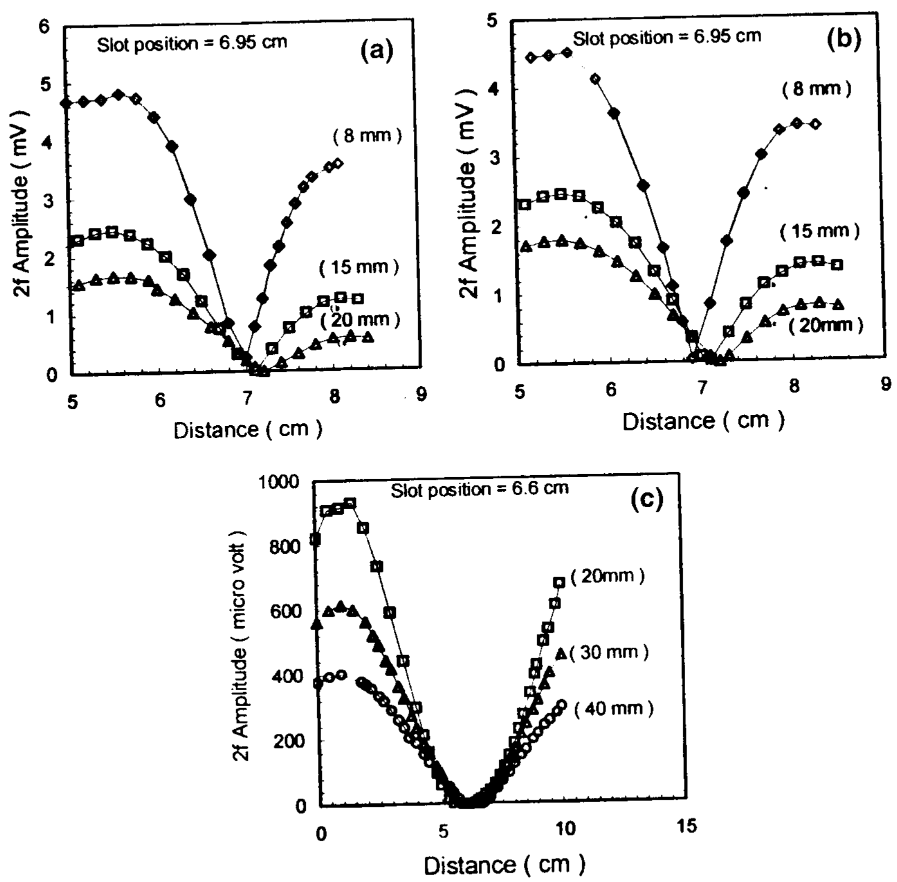

Figure 5. Variation of the second harmonic signal when the superconducting BPSCCO sensor is scanned over ferromagnetic plates having slots of width (**a**) 0·9 mm, (**b**) 2·5 mm and (**c**) 5 mm, respectively for different stand-off distances.

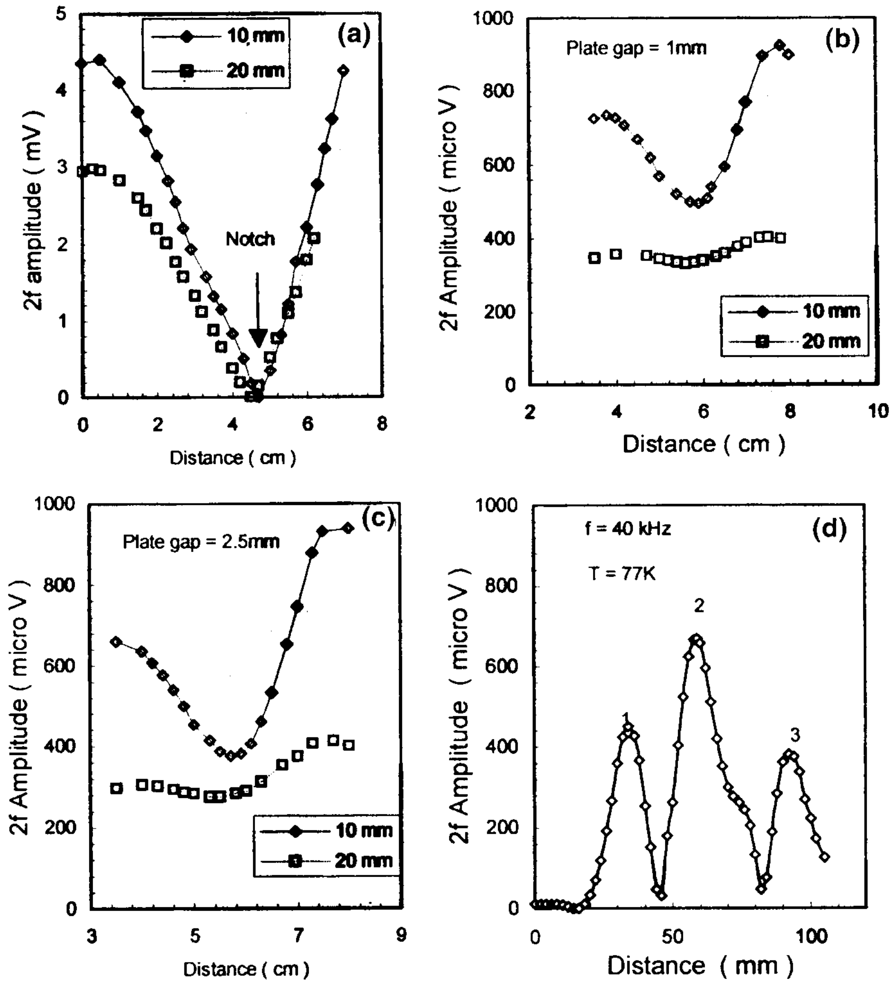

Figure 6. Variation of the second harmonic signal when superconducting BPSCCO sensor is scanned over (**a**) a ferromagnetic plate having notch at a distance of 4·5 cm, (**b**), (**c**) ferromagnetic plates separated by gaps of 1 mm and 2·5 mm, respectively for different stand-off distances, and (**d**) three ferrite pieces embedded in a (Pb–Sn) bar.

region in the test piece passes underneath the BPSCCO sensor. It may be noted that as the slot width increases, the signal tends to become broader. Figure 5 also shows the sensor response of the same test pieces for different stand-off distances. For a stand-off distance of 8 mm, a sharp resolution and detection of the slot at the exact location on the test pieces is very clearly evident. As the stand-off distance increases, the area of field distortion becomes larger and consequently, the amplitude of the signal decreases and some broadening in the response takes place. Figures 6a–c show the results of the scan for different stand-off distances for a test piece containing a notch and for the case in which two ferromagnetic plates are kept separated by various distances, respectively. In both the cases, position of the notch in the plate, as well as, the gap between the two plates could be clearly detected.

Experiment to check the feasibility of detection of small magnetic inclusions in a non-magnetic matrix by the present NDE device using the same sensor was carried out on a suitably prepared test piece. Three small ferrite pieces ($\phi \sim 1{\cdot}5$ mm) were buried at a depth of ~ 5 mm from the top surface in a 150 mm × 25 mm × 15 mm (Pb–Sn) solder bar at accurately known location. The test piece thus prepared was scanned along the length of the test piece for a stand-off distance of 10 mm. Figure 6d shows the variation in the amplitude of the sensor's $2f$ signal as a function of position of the sensor on the test piece. Three peaks observed in figure 6d correspond well to the known locations of the ferrite pieces buried in the non-magnetic test piece. Differences in the magnitude of the peak values observed are likely to be due to the differences in the magnetic field strength of the ferrite pieces used.

4. Conclusions

A sensitive polycrystalline BPSCCO magnetic sensor based on its linear and reproducible second harmonic response in low d.c. field has been fabricated. When operated at 40 kHz under an a.c. field of 16 G at 77 K, a noise sensitivity of ~ $3{\cdot}16 \times 10^{-9}$ T/$\sqrt{\text{Hz}}$ is attained. Feasibility of application of this sensor in non-destructive testing of various types of defects in ferromagnetic materials, as well as, for detection of magnetic inclusions in non-magnetic matrix has been demonstrated. It is possible that such a $2f$ response based BPSCCO magnetometer housed in a miniature cryostat and complete with inexpensive front-end integrated electronics and display will provide a novel NDE device. Work in that direction is now in progress and will be reported soon.

Acknowledgements

The financial support received in the form of project funding by the Department of Science and Technology, New Delhi, is gratefully acknowledged.

References

Braginski A I and Krause H J 2000 *Physica* **C335** 179

Buckley J R, Khare N, Donaldson G B, Cochran A and Hui Z 1991 *IEEE Trans. Magn.* **27** 3051

Dey T K, Chattopadhyay M K and Ghatak S K 1999 *Sensors and Actuators* **B55** 222

Gallop J C, Lilleyaman S, Langham C D, Redcliffe W J, Gee M and Stewart M 1988 *Cryogenics* **28** 691

Gallop J C, Lilleyaman S, Langham C D, Redcliffe W J and Stewart M 1989 *IEEE Trans. Magn.* **25** 896

Ghatak S K, Mitra A K and Sen D 1992 *Phys. Rev.* **B45** 951

Gielisse P J, Niculescu H, Roy B, Jones K W, Larkin G and Hu Z 1991 *Supercond. Sci. Technol.* **4** 416

Ishida T and Goldfrab A B 1990 *Phys. Rev.* **B41** 8937

Khare N, Gupta A K, Srivastava S K, Khare S and Padmanaban V P N 1997 *Meas. Sci. Tech.* **8** 29

Kim Y, Lam Q H and Jeffries C D 1991 *Phys. Rev.* **B43** 11404

Lam Q H and Jeffries C D 1989 *Phys. Rev.* **B39** 4772

Muller K H 1990 *Physica* **C168** 585

Muller K H, Macfarlane J C and Driver R 1989 *Physica* **C158** 366

Navarro R, Lera F, Rillo C and Batolome J 1990 *Physica* **C167** 549

Qin M J and Ong C K 1999 *Phys. Rev.* **B61** 9786

Xenikos D G and Lamberger T R 1990 *Phys. Rev.* **B41** 869