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Single-sided mobile NMR with a Halbach magnet

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Abstract

A single-sided mobile NMR apparatus with a small Halbach magnet was constructed for the first time. It is lightweight, compact and exhibits good sensitivity. The weight of the device is only 2 kg, and the NMR signal of the pencil eraser block can be detected in one shot using the device. This study describes the characteristics of this instrument, including the profile of static magnetic flux density, B_0 , the sensitivity in the depth direction and its effectiveness in one-dimensional profiling. Its usefulness in differentiating soft materials and evaluating the extent of damage of a material is demonstrated based on T_2 relaxation data. The moisture absorbance also can be observed from the increase of the echo amplitude of the NMR spin echo signal.

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1. Introduction

The single-sided NMR has drawn much attention recently because this instrument is more flexible than conventional bore-shaped magnets under some circumstances. Briefly, single-sided NMR devices are designed to produce a moderate homogeneous static magnetic flux density, B_0 , outside the magnet, where radio frequency (RF) excitation and signal detection are performed. This instrument can be attached to virtually any sample and detect an NMR signal outside the magnet; hence, the sample size is unlimited. A small and portable single-sided NMR apparatus can facilitate in situ NMR experiments, especially on immobile samples, such as wet rocks [1], plants and frescos [2]. Some researchers have also employed singlesided mobile NMR to control the quality of soft materials or food systems [3-7]. The single-sided NMR magnets typically have lower signal sensitivity and spectral resolution than conventional bore-shaped magnets, but these shortcomings have gradually been overcome [8].

The design of a single-sided mobile NMR instrument is critical. For over about a decade, various magnets have been developed, such as the horseshoe magnet [9,10], the singlebar magnet [11], the magnet array [12,13], the barrel magnet [14] and the four-block magnet [15]; the magnets are carefully designed to exhibit different characteristics such as the profiles of B_0 , the detection range and the size. In this work, a different strategy is used: a compact conventional NMR magnet is built first, and then its stray field outside the magnet is directly used as the polarization source B_0 of a single-sided mobile NMR device. The magnetic flux density close to the magnet edge should have enough strength and sufficient homogeneity, for the use of single-sided mobile NMR.

Several researchers have recently applied Halbach magnets [16] to build NMR magnets, for use in a tabletop NMR [17], a mobile (but not single-sided) NMR for measuring drilled rock [18,19] and prepolarizing samples in the earth-field NMR experiments [20]. A Halbach magnet generates a highly homogeneous static magnetic flux density near its center and a moderately homogeneous static magnetic flux density near its top; it can also be built to be very compact. Accordingly, a single-sided mobile NMR with a small Halbach magnet was constructed; the sensitive region of this instrument is near its top. Finite-element

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simulations were performed using Maxwell 3D commercial software (Ansoft Co., Pittsburgh, PA, USA). The results showed that the field homogeneity on the top of the Halbach magnet was better than that of the horseshoe magnet [9]; this fact motivated the implementation in this study. Several measured properties of our single-sided mobile NMR Halbach magnet are discussed, and several associated applications are presented herein.

2. Materials and methods

The scheme of our Halbach magnet herein is based on that of Moresi and Magin [17], who used eight radial-magnetized, cylindrical $SmCo_{17}$ magnets to form



Fig. 1. (A) Top view of the completed magnet structure and coordinate definition. Eight cylindrical magnets synthesize a B_0 on the top of the magnet. The direction of B_0 near the top of the magnet is parallel with the top surface of the magnet assembly; this direction is defined as the *z* direction. (B) Hollow, infinitely long magnetized cylinder, illustrating the ideal Halbach structure. The figure is based on Ref. [21].

their Halbach magnet. In this study, the design is shortened to render the entire magnet more portable, and NdFeB cylindrical magnets (diameter: 2 cm, height: 5 cm, from Ming-Yen Electronics, Taichung, Taiwan) are used to increase the magnetic flux density strength. The assembling process is similar to that described elsewhere [17]. Specifically, more cylindrical magnets were bought in advance, and the eight whose surface magnetization values were closest with each other, as measured by a Hall probe (Model 7010 Gauss/Teslameter, F. W. Bell Co., Orlando, FL, USA), were chosen. After the poles of those magnets were marked, a small, milled aluminum piece was attached to the bottom of each magnet, using high-quality 3M DP810 epoxy resin (3M Co., St. Paul, MN, USA). A machined polyoxymethylene plastic steel holder (diameter: 10 cm, length: 6 cm, from Hon-Ku Trading Co., Taipei, Taiwan) was made to fix the magnets, such that the centers of the cylindrical magnets were equally spaced and were 3 cm from the center of the holder in the transverse plane. A 3-cm-diameter hole was drilled through the center axis of the holder to accommodate the RF parts. The glued aluminum pieces were clamped using pliers; the magnets were then inserted into the holder and rotated to their desired angles. The cylindrical magnets were fixed in the holder with stainless steel bolts pressed from the lateral sides of the holder. Fig. 1A depicts the complete structure. The eight magnets in the holder comprise a Halbach configuration: if the center of the cylindrical magnet makes a polar angle φ with the holder's center, then the direction of the cylindrical magnet's magnetization is made to point at a polar angle of 2φ ; the synthesized magnetic field in the bore region will then be quite uniform and point in the $\varphi = 0$ direction. Ideally, consider a hollow, infinitely long cylinder made of magnetic material with an inner radius of r_1 and an outer radius of r_2 (Fig. 1B). If it has magnetization \vec{J} [21]:

$$\vec{J} = J_0 \Big[(\cos\varphi) \vec{r} + (\sin\varphi) \vec{\varphi} \Big], \tag{1}$$

then the magnetic flux density $\rightarrow B$ in the $r < r_1$ region will be:

$$\vec{B} = J_0 \left(\ln \frac{r_2}{r_1} \right) \left[(\cos \varphi) \vec{r} - (\sin \varphi) \vec{\varphi} \right].$$
⁽²⁾

This field is uniform and points in the $\varphi = 0$ direction. The structure of our magnet is an approximation to the ideal distribution in Eq. (1). The stray field in the near region above the top of the magnet assembly is used to perform single-sided mobile NMR experiments. The direction of B_0 near the top of the magnet assembly is horizontal (the *z* direction in Fig. 1A); consequently, the RF coil can be built easily. The weight of the magnet assembly is 1.5 kg. All cylindrical magnets have tolerances; hence, the magnetic field homogeneity is tuned manually by slightly adjusting the polar angle or the vertical position of each cylindrical magnet and checking the Hall probe's readings at some

particular points on the top of the magnetic assembly, until the homogeneity could no longer be improved.

The magnetic flux density on the center of the top of the magnet, which was determined by measuring the NMR spin echo signal of a $10 \times 10 \times 0.12$ mm paraffin sheet, is around 0.2643 T or 11.25 MHz as a ¹H resonant frequency, at 26°C. A spin echo sequence acquired from $\theta_x - \tau - 2\theta_y - \tau$ with θ_x and $2\theta_y$ pulses with the same width but different amplitudes was used throughout this work. If the ambient temperature changes, the corresponding magnetic flux density value can be determined from the known temperature coefficient of the NdFeB magnets (-0.13%/°C) or simply by directly measuring the NMR signal of a thin sample.

The RF probe consists of an RF coil and a tuning-andmatching circuit. The RF coil is a double-sided, three-turn, counter-wound planar spiral coil manufactured on a standard, 1.6-mm-thick printed circuit board (PCB; Kinsten Industrial Co., Taipei, Taiwan). The coil is 1.8 cm long and 1.6 cm wide; the conductor width is 0.5 mm. The doublesided layout increases the coil's inductance, enabling the capacitance value for resonance to be reduced. The counterwound pattern can make the coil more sensitive to the spatial gradient of the RF field, enhancing the coil's resistance to the radiation noise [22,23]. The layout of this RF coil is similar to that described by Anferova et al. [23]. The resistance and inductance of this coil measured at 0.5 MHz are 0.45 Ω and 0.75 μ H, respectively. The coil is placed in the magnet bore, with its upper surface 0.5 mm below the top surface of the magnet assembly, so that the coil does not block the sample. The magnetic flux generated by this coil is from one spiral center to the other; thus, the RF coil is orientated such that a straight line between the two spiral centers is perpendicular to B_0 .

In the original design, the tuning-and-matching circuit was housed in an $11.5 \times 5.6 \times 8.9$ cm aluminum box, which is placed below the magnet assembly (Fig. 2A). The coil and the tuning-and-matching circuit were connected by leads on a PCB stem. The tuning part includes a 60-pf nonmagnetic chip capacitor (American Technical Ceramics Co., New York, USA) and three 120-pf nonmagnetic variable capacitors (Voltronics Co., Denville, NJ, USA) in parallel; the matching part is a 120-pf nonmagnetic variable capacitor. With this arrangement, the resonant frequency of the RF coil can sweep from 13.4 to 7.15 MHz. This probe was employed only to measure the signal sensitivity of our system in the depth or y direction (direction is defined in Fig. 1A) since this experiment must cover a large frequency range. When the probe resonates at 11 MHz, the bandwidth of the probe measured by a method described elsewhere [23] is 0.54 MHz, which corresponds to a quality factor Q of 20.4. The probe operates in a space with a strong B_0 gradient (see the next section); hence, the quality factor of the RF probe need not be very high to enable a signal to be collected from many locations. This RF probe weighs 0.4 kg.





Fig. 2. (A) Original version of RF probe. The bottom variable capacitor is a matching capacitor, and the other three variable capacitors are for tuning. (B) Compact version of RF probe, which is inside a copper tube and inserted into the bore of the magnet. The structure below the magnet is a support because tuning and matching are performed from the bottom of the magnet.

A compact version of the RF probe, which is more mobile and easier to carry, was also made. The RF coil and the matching circuit of this probe are the same as those used in the original probe, but the tuning circuit of this probe only consists of a 270-pf nonmagnetic chip capacitor and a 120-pf nonmagnetic variable capacitor in parallel. The tuning-and-matching circuit is consequently small enough to be integrated into a copper tube, which can be inserted into the center hole of the holder (Fig. 2B). The upper face of the coil is 0.4 mm below the top of the magnet; the Q of the probe is 18.3 when it resonates at 11 MHz. The remaining experiments were conducted using this probe. This RF probe weighs 0.5 kg.

A MARAN DRX spectrometer (Oxford Instruments Co., Abingdon, Oxon, UK) and a 300-W pulse mode RF amplifier



Fig. 3. (A) Measured B_0 profile on the top surface of the magnet (lower right), 0.5 mm above the top surface of the magnet (upper right) and 1 mm above the top surface of the magnet (upper left). The measured area is 20×20 mm. (B) Measured B_0 profile on the *xy* plane (upper left) and *yz* plane (upper right) above the top surface of the magnet. The measured area is 20 mm (width)×10 mm (height).

were employed to perform the NMR experiments. A 500-W RF amplifier (Model: BT00500-CL, Tomco Electronics, Norwood, SA, Australia) was used only when measuring the signal sensitivity in the y direction. Several modified T/R switches (originally from Bruker Co., Rheinstetten, Germany) were employed to duplex the signals.

3. Results

3.1. Static magnetic flux density profile

The static magnetic flux density profile on and above the top of the magnet was measured using a Hall probe fixed on a home-assembled 3D translation stage. Measured field



Fig. 4. Gradient magnitude $|\Delta B_z/\Delta y|$ along y-axis.

profiles were plotted using MATLAB v 6.5 (Mathworks Co., Natick, MA, USA). The origin of the measurement was the geometrical center of the top of the magnet; the coordinate definition is defined in Fig. 1A. The measured static magnetic flux density on the transverse plane (xz plane) was most homogeneous at about 0.5 mm above the top (Fig. 3A). The field profiles become curved and the field strength decays as the vertical (y) distance increases, as can be observed from the contour lines on the xy and yz planes (Fig. 3B). These field plots indicate that the observed sample should be placed close to the top of the magnet, where the magnetic flux density is most intense and homogeneous.

A field gradient in the y direction (Fig. 4) is 14.1 T/m at the top of the magnet. This gradient strength is nearly constant for 0 mm < y < 2 mm. A 1-mm-thick sample has a frequency span of 0.6 MHz at this field gradient. Therefore, this field gradient can be exploited to resolve slices with a thickness of several tens of microns. A one-dimensional profiling experiment will be presented later.

3.2. Signal sensitivity in the y direction

The sensitivity map in the *y* (depth) direction from the origin was determined by measuring the time domain spin echo signal amplitude of a $10 \times 10 \times 1$ mm pencil eraser sheet that was moved along the *y*-axis. The original RF probe, which has many tuning capacitors, and a 500-W RF amplifier were used to perform this experiment. Fig. 5A shows the measured results. Two RF excitation levels were used (50% and 100% of the RF amplifier rated output voltage; please refer to Fig. 5A caption). Results indicate that although the use of a high-voltage RF excitation helps to increase the NMR echo amplitude when the sample is far away from the top of the magnet, the signal amplitude is greatest when the sample is closest to the magnet. Therefore, the proposed single-sided NMR is a surface-type detector. Furthermore, if a pencil eraser block is placed directly on

the top of the magnet, the NMR spin echo signal can be detected in a single shot (Fig. 5B). The instrument's sensitivity is satisfactory.

3.3. Profiling thin layers in the vertical direction

The gradient $|\Delta B_z/\Delta y|$ along the *y*-axis calculated from the measured data is nearly a constant within a vertical distance of 2 mm from the top of the magnet. Although this gradient strength varies in the horizontal direction, if the sample's (horizontal) area is maintained sufficiently small, then the effect of the horizontal B_0 and gradient inhomogeneity can be reduced and one-dimensional profiling experiments can be performed in the *y* direction. In this field



Fig. 5. (A) Sensitivity map in the *y* direction. The diamond points were obtained using half of the rated output voltage of the RF power amplifier for excitation; the circular points were obtained using the full rated RF output voltage for excitation. The number above each peak is the distance between the top of the magnet and the sample. The following parameters were used: RF pulse length=4 μ s, τ =100 μ s, recycle delay=0.1 s and scan time for each point=10 min. (B) Spin echo signal of a pencil eraser block obtained in a single shot. The following parameters were used: central frequency=11 MHz, RF pulse length=2 μ s and τ =50 μ s.



Fig. 6. (A) Magnitude spectrum of the NMR spin echo signal of three water layers. The following parameters were used: central frequency=11 MHz, RF pulse length=2 μ s, τ =80 μ s, recycle delay=0.1 s and total scan time= 5 min. (B) Magnitude spectrum of the NMR spin echo signal of four water layers. The following parameters were used: central frequency=11.02 MHz, RF pulse length=2 μ s, τ =80 μ s, recycle delay=0.1 s and total scan time=5 min. Five CPMG echoes were added before the Fourier transformation to increase the SNR.

gradient, a 1-mm-thick sample has a 0.6-MHz frequency span; thus, the whole sample can be excited if its thickness is less than several hundred micrometers. The sample was composed of several 0.02-M CuSO₄-doped, 0.05-mm-thick water layers prepared with a micropipet. The water layers were separated by 0.1-mm-thick transparency films. The horizontal area of the sample was 10×10 mm. Three or four such water layers were placed horizontally on the top of the magnet; one or two cover glasses were inserted below the sample to adjust the height of the center of the sample close to y=0.5 mm, where B_0 is most homogeneous in the transverse direction. A 2-µs RF pulse and an echo time of 170 µs were used herein; 128 points were acquired and a Fourier transform was used to resolve the peaks of the water layers at different resonant frequencies. Both experiments were completed in 5 min. In the case of four water layers, five Carr–Purcell–Meiboom–Gill (CPMG) echoes were added before Fourier transformation to increase the signalto-noise ratio (SNR) [15] because the edge layers were considerably off-resonance and had lower signal intensities. In both results (Fig. 6A and B), the water peaks of different layers can be distinguished. The interpeak distances were calculated from the spectra and the measured gradient value; the calculated distances were then compared with the actual interpeak distance (0.15 mm). The error was within 8% in the case of three water layers. The maximal error was 13% of the actual distance in the case of the four water layers.

3.4. Applications of nondestructive evaluation

Single-sided mobile NMR can be used in nondestructive evaluation by probing the relaxation state or measuring the water content of the material, as demonstrated in the following three experiments. In the first experiment, the developed instrument was used to distinguish two polymers, a 0.65-mm-thick nylon sheet and a 1-mm-thick polypropylene (PP) sheet, based on their T_2 relaxation curves obtained by recording spin echo signal amplitudes at different echo times. A CPMG sequence was not used because such a sequence in inhomogeneous B_0 and B_1 fields cannot be used to evaluate T_2 accurately [3,24]. The on-resonance plane was established 0.5 mm above the top of the magnet, and the coil's upper surface was 0.4 mm below the top of the magnet. In the case of a 2-µs RF pulse length, the coil's epoxy substrate did not yield background signals at an echo time of 40 µs, which was the shortest echo time used herein. Each measurement point took 5 min; five measurements were made and the standard deviations were plotted. The T_2 values were fitted using Origin v7 (OriginLab Corp., Northampton, MA, USA). The single-sided NMR measurements revealed that the T_2 constant of the nylon sheet $(22.42\pm0.18 \text{ } \mu\text{s})$ was less than that of the PP sheet $(427.58\pm20.81 \ \mu s)$; single-sided NMR can clearly distinguish between these two materials (Fig. 7A).

The second experiment distinguished wood that underwent various degrees of thermal damage through singlesided mobile NMR. A 100×90×2.7 mm dry wood plate was burned by natural gas fire, and the NMR T_2 relaxation data of the site after different degrees of damage (0, 2, 4 and 12 s of fire burning) were measured. The experimental method was the same as that used in the first experiment. The results showed a clear correspondence between the extent of thermal damage and the echo amplitude decay (Fig. 7B). The fitted T_2 value was $39.27\pm2.25 \ \mu s$ for the intact wood plate. After the wood plate underwent 2, 4 and 12 s of fire burning, the fitted T_2 value of the wood plate became 37.56 ± 1.26 , 35.67 ± 1.18 and 32.85 ± 0.5 µs, respectively. The T_2 value also decreased as the degree of thermal damage was increased, indicating that the damaged site also has lower proton mobility.

The third experiment used single-sided mobile NMR to measure the moisture absorbed by a round cracker (diameter: 5.4 cm, thickness: 3 mm). First, the NMR spin echo signal of a piece of cracker inside its intact plastic package was measured at an echo time of 40 μ s. The plastic



package was thin (thickness: 0.06 mm) and contributed negligibly to a signal; hence, the NMR signal of the cracker could be measured without removing the package. The package was then cut open at one edge. The cracker (still inside the opened package) was placed in a covered beaker; the temperature and humidity inside the beaker were controlled between 22° C and 24° C and between 48% and 58%, respectively. The NMR spin echo signal of the cracker was measured daily at an echo time of 40 µs (Fig. 7C). Another piece of cracker inside its intact package was also measured daily and served as a control group. The increase of the echo amplitude of the cracker with the opened package clearly showed the water absorbance of the cracker. This experiment demonstrates the potential application of the instrument in the food industry.

4. Discussion

The Halbach scheme was originally designed for conventional bore-type NMR; hence, the existing structure should be modifiable and its performance as a single-sided NMR device can be improved. For instance, if another magnet is present in the bore region, its orientation, size and vertical position can be adjusted to control the field strength, homogeneity, field gradient above the top of the magnet and the detection range. Pilot simulations indicate that this approach is feasible. More finely adjusting the position of the surrounding eight magnets or adding shim pieces [25] increases the horizontal field homogeneity. Sixteen magnets can be used instead of eight to increase the horizontal field homogeneity [18]; differently shaped magnets can also be employed to increase the field strength or homogeneity. However, the manufacturing process may be more time consuming. Finally, ideas should be borrowed from other single-sided magnet designs to improve the performance of the proposed instrument.

Fig. 7. (A) NMR T_2 relaxation decay curve of nylon and PP sheets measured by single-sided NMR. NMR spin echo amplitude at each point was measured five times and standard deviations were plotted. The standard deviations are smaller than or equal to the size of the marker. The following parameters were used: central frequency=11 MHz, RF pulse length=2 µs, recycle delay=0.1 s and scan time=5 min for each point. (B) NMR T_2 relaxation decay curve of the intact and burned dry wood plate. The following parameters were used: central frequency=11 MHz, RF pulse length=2 µs, recycle delay=0.1 s and scan time=5 min for each point. NMR spin echo amplitude at each point was measured five times and standard deviations were plotted. (C) Measured NMR echo amplitudes at an echo time of 40 µs from two pieces of crackers. The plastic package of one cracker was cut open and moisture was absorbed by the cracker. Another cracker inside the intact plastic package was the control group. The following parameters were used: central frequency=11 MHz, RF pulse length=2 µs, echo time=40 µs, recycle delay=0.1 s and scan time=5 min for each point. NMR spin echo amplitude at each point was measured five times and standard deviations were plotted.

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A single-sided NMR instrument with a small Halbach magnet was built. The most homogeneous region of the stray field is very close to the top of the magnet, where samples can be placed to collect the NMR signals. The sensitivity of the NMR instrument is satisfactory, and onedimensional profiling experiments can be performed on thin layers on the magnet. Application experiments demonstrate that the developed single-sided mobile NMR instrument can be used conveniently in nondestructive evaluation of samples of unlimited size. Several modifications to the proposed instrument are feasible; applying these modifications to improve the performance of the instrument is our current work. Other single-sided NMR designs can be combined to improve the capability of this instrument.

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