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AUTOMATION SYSTEMS IN SCIENTIFIC RESEARCH AND INDUSTRY

Pulsed Nuclear Magnetic Resonance Magnetometer

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Abstract—A precision magnetometer based on pulsed nuclear magnetic resonance (NMR) is described. This magnetometer measures constant magnetic fields with an absolute error not more than 10^{-6} and a resolution of up to 10^{-7}. The use of modern digital technologies, such as FPGA, made it possible to significantly accelerate and optimize NMR signals. The magnetometer structure is and the signal processing methods are described, measurement errors are analyzed, and experimental data are presented.

Keywords: NMR magnetometer, FPGA, NMR sensor, magnetic fields, digital signal processing.

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INTRODUCTION

Nuclear magnetic resonance (NMR) magntetometers are used for measurements of the Larmor frequency of nuclear precession ($\omega_{\rm pr}$) in a magnetic field, which is related to the field induction B through the gyromagnetic ratio γ, which is the property of this type nucleus: $\omega_{\rm pr} = \gamma B$. The standard method used in most NMR magnetometers [1] manufactured in the world is an autodyne method based on the use of a self-oscillator whose frequency is automatically tuned to the NMR frequency. One of the main disadvantages of this method is that the sensor is part of the resonance circuit, which significantly reduces the field measurement range covered by one sensor and places an upper limit on the working frequency; thus, one has to measure high fields (more than 1 T) by using lithium and deuterium nuclei as working nuclei, rather than protons giving a stronger signal. Measurement of magnetic fields in a wide range, e.g., from 0.1 to 2 T, requires replacing five to six NMR sensors, which is not always possible when working with electrophysical installations. The fact that the sensor is part of the resonance circuit also places a limit on the distance between the sensor and electronics, which should not be more than 0.5 to 1 m.

Pulsed methods are widely used in NMR spectroscopy and NMR tomography [2]. They have been successfully used in the construction of NMR magnetometers at the Budker Institute of Nuclear Physics, Siberian Branch, Russian Academy of Sciences (BINP SB RAS) for the last 20 years. The choice of pulsed methods was mainly due to the requirements for their operation in electrophysical installations at the BINP SB RAS: it is required that one sensor cover a wide field range and be located at a distance of 10 m from the electronics. This significantly increases the complexity of the magnetometer electronics, but this factor has almost ceased to be governing because of the fast progress in the area of the component base.

The purpose of this work is to create a new compact magnetometer based on pulsed NMR methods, in which modern digital technologies would significantly accelerate and optimize the signal processing process and eventually provided a measurement resolution of the order of 10^{-7}.

MEASUREMENT METHOD

The magnetometer described is based on the pulsed technique widely used in NMR spectroscopy and presented in Fig. 1. The essence of this technique is that the nuclei contained in the sample are subjected

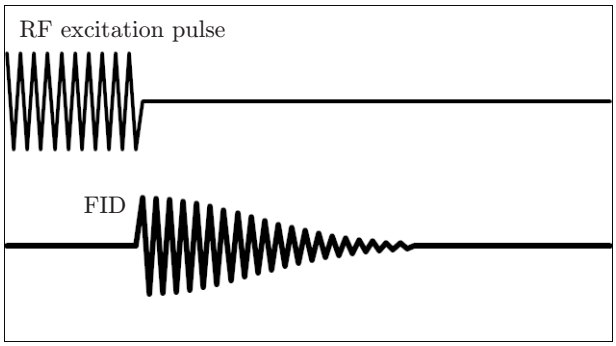

Fig. 1. Nuclear magnetic resonance technique underlying the NMR magnetometer.

to a radio frequency (RF) pulse with subsequent recording of the free induction decay (FID) signal [3]. The duration of the signal affecting the field measurement accuracy is determined by two factors: the transverse relaxation time T_2 due to the interaction of the spins of nuclei with each other and with the lattice and the variation in the frequency of nuclear precession in the sample due to the nonuniformity of the magnetic field. The the RF excitation pulse frequency ω_{exc} should be close to the nuclear precession frequency ω_{pr}. To obtain the required FID signal amplitude, the frequency difference $\omega_{\text{exc}} - \omega_{\text{pr}}$ should be smaller than $1/\Delta T_{\text{exc}}$, where ΔT_{exc} is the duration of the RF excitation pulse. The RF pulse duration, in turn, should make the magnetization vector of the nuclei rotate by an angle θ approximately equal to 90° (in this case, the signal is maximal): $\theta = \gamma b_{\text{exc}} \Delta T_{\text{exc}} \approx \pi/2$, where b_{exc} is the RF excitation field amplitude in the sample. The characteristic values of ΔT_{exc} lie in the range from 3 to 50 μs and depend on the induction of the measured field, its uniformity, and the sensor sample volume. During one measurement cycle, the sample is usually excited many times with acquisition of NMR signals, and this increases the accuracy of measurements. The signal acquisition rate (or the frequency of repetition of excitation pulses) depends on the recovery rate of the stationary nuclear magnetization of the sample, which is defined by the time of transverse relaxation T_1 due to the interaction of the nuclear spins with the lattice.

NMR SENSORS

The sensors used in pulsed NMR magnetometers have a very simple structure. They comprise a sample containing working nuclei, coils wound around the sample, and a housing. The utmost simplicity of the sensors makes it possible to place them in housings of very small size. Thus, we developed a 0.6 mm thick sensor for the measurement of magnetic fields in narrow gaps. Due to the absence of resonance elements, one sensor can cover a wide field range and the value of $B_{\max}/B_{\min}$ can achieve several tens. Another advantage is the possibility of placing the sensor at a long distance (up to 10 m and farther) from the electronics.

The working substances used in this study are water, rubber, and aluminum powder. Water provides the largest signal/noise ratio. Rubber, in which the working nuclei include protons, have a serious disadvantage such as the small transverse relaxation time T_2, which significantly reduces the accuracy of measuring uniform fields. Metallic aluminum powder is used to measure magnetic fields at ultralow temperatures, such as liquid helium temperature [4].

STRUCTURE AND OPERATING PRINCIPLE OF THE NMR MAGNETOMETER

A diagram of the four-channel NMR magnetometer is shown in Fig. 2. The magnetometer allows simultaneous measurements with four sensors. It consists of the main electronics unit (Fig. 3) placed in a euromechanical housing of height 1U, four extension preamplifiers, and four sensors. Multichannel operation is provided by alternate connection of the preamplifiers to the main unit using special multiplexers.

The functional components of the magnetometer are a two-channel frequency synthesizer, a transmission path, and a reception path. The transmission path generates RF excitation pulses of an amplitude of 15 V (pulse power of nearly 2 W), which are fed to the NMR sensor coil, which is simultaneously a receiving coil. The RF excitation pulse generator is very simple. A RF pulse of a rectangular shape with an amplitude of 2.5 V, and a synthesizer frequency of 1 is formed using the CMOS logic,. This pulse is sent to an IXDD415SI driver, capable of driving a large capacitive load (up to 5 nF) and provide an RF voltage amplitude of up

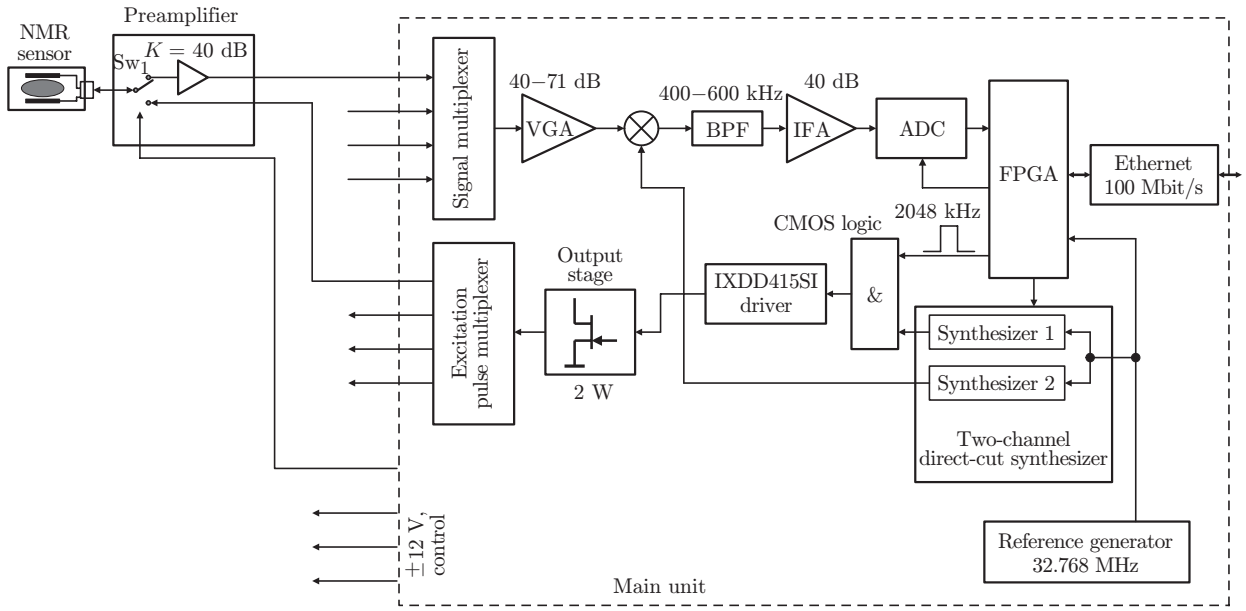

Fig. 2. Block diagram of the NMR magnetometer.

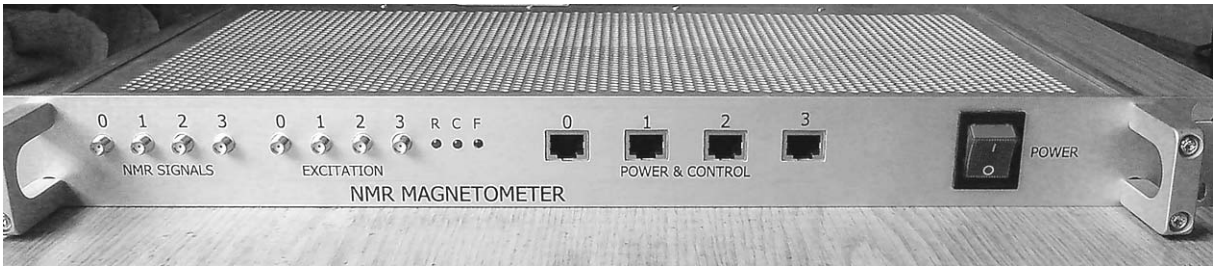

Fig. 3. Main unit of the magnetometer (view from the front panel).

to 5 V on the gate of a KP907A output transistor. The output transistor operates in a cut-off mode. It is locked by default, but it can be unlocked by incoming RF voltage. The output pulse power is around 2 W, and the average consumed power of the entire RF pulse generator during measurements, taking into consideration the high RF pulse ratio (1000–10 000), is very small (0.2–2 mW).

During reception, the FID signal induced at the coil ends by the nuclei is sent through a switch sw_1 to the input of a low-noise amplifier with a transmission coefficient of nearly 40 dB. Then the signal is transmitted through the cable connecting the preamplifier with the main unit into the signal multiplexer and arrives at a variable gain amplifier (VGA), whose transmission coefficient is programmable in the range from 40 to 71 dB. As the NMR signal amplitude increases with the field, control of the transmission coefficient allows maintaining an optimal signal level within the entire measured field range. The reception path of the magnetometer operates in a superheterodyne mode with conversion into an intermediate frequency. After the VGA, the signal is mixed with the reference signal from synthesizer 2 whose frequency F_2 is about 500 kHz smaller than that of synthesizer 1. After the mixer, the NMR signal with a frequency close to 500 kHz is fed through a bandpass filter (BF) to an intermediate frequency amplifier (IFA) and is then converted into digital form by a 12-bit ADC with a 2048 kHz sampling frequency. Further processing of the NMR signal is carried out in digital form in an FPGA (Altera) circuit; it includes primary signal processing (subtraction of the constant component) and finding the signal spectrum using a Fourier transform. The final signal processing occurs in a computer, which reads the signal spectrum from the unit and finds the middle of the integral of the resonance peak ΔF_p, which is the difference between the NMR frequency F_NMR and the frequency F_2. The sough NMR frequency is found as the sum of the determined ΔF_p and known F_2 frequencies.

The nuclei excitation and FID signal reception processes are carried sequentially in time. The transition from the excitation stage to the signal reception stage is provided by a switch sw_1. There is a large difference between the excitation voltage level and the NMR signal level, which is nearly 80 dB. The "dead" time, i.e., the period from the end of the RF excitation pulse to the moment when the reception path is ready to receive very small FID signals (characteristic amplitude of these signals are microvolt units), should be as small as possible. This allows recording rapidly attenuating signals which occur in the cases of large nonuniformity of the measured fields or when using sensors based on metal powders having a small transverse relaxation time. In the described magnetometer, the dead time is approximately 5 μs.

The magnetometer is connected to an Ethernet switch at 100 Mbit/s and can be accessed from any computer connected to this network.

OPERATION MODES

An NMR signal is induced at the ends of the sensor coil if the difference between the nuclear precession frequency and the excitation frequency ($\omega_{\text{pr}} - \omega_{\text{exc}}$) does not exceed the value of $\sim(1/\Delta T_{\text{exc}})$. If the measured field is not known in advance with an accuracy of the order of $1/(\gamma \Delta T_{\text{exc}})$, the magnetometer is provided with a search mode, where the excitation and reference signal frequencies are scanned in a given range to seek the signal peak. If the peak exceeds some threshold value, it is concluded that the excitation frequency corresponding to this peak is the approximate value of the sought NMR frequency. Next, the measurement mode is activated to find the exact value of the NMR frequency, and the excitation frequency follows the NMR frequency.

DIGITAL SIGNAL PROCESSING

The most time-consuming part of the signal processing — calculation of their spectra — is carried out in the FPGA of the magnetometer base unit.

Actually, a digital spectrum analyzer is implemented in the FPGA. The source data are a signal time array of 16384 points, obtained using a ADC with a sampling frequency of 2048 kHz. The following spectrum parameters are defined: the center of the frequency range F_0, the frequency range D, and the frequency step ΔF. The digital frequency synthesizer is retuned in the range from $F_0 - D/2$ to $F_0 + D/2$ with a step ΔF, and the amplitude of the spectrum $S(F)$ is found for each frequency F. The obtained signal spectrum is further transmitted to a computer where it undergoes final processing. The magnetometer provides an automatic tuning of the parameters of the digital spectrum analyzer for the real NMR signal spectrum.

The whole process of digital signal processing consists of two phases: intermediate and main. During the intermediate phase, we roughly find the NMR frequency F_{r} and calculate the approximate width of the signal spectrum ΔF_{sp}, the results are used to estimate the NMR signal duration, and the optimal filter parameters are selected. It is known that the effect of amplitude noise on the measurement results can be minimized by filtering the signal through a "coherent" filter [5] by multiplying the signal into a function that coincides with the signal envelope. To avoid large losses, a rectangular window is used as this function; i.e., out of the whole array, we take the number of signal points N that contain $\sim$90% of the signal energy. The main phase results in a spectrum with a maximum frequency resolution with the center of the range F_{r} and the size of the range $D \sim 3\Delta F_{\text{sp}}$. Further, we find the middle of the integral of the resonance peak ΔF_{p}, which is the difference between the NMR frequency $F_{\text{NMR}}$and the frequency of the synthesizer F_2. The whole cycle of signal processing takes 20–40 ms, which is comparable to the excitation pulse period.

SIGNAL ACQUISITION

To increase the signal/noise ratio and improve the accuracy of measurements, the magnetometer offers an opportunity of signal acquisition by summing the signal arrays obtained after various excitation pulses. An increase in the signal/noise ratio is achieved at the cost of increasing the measurement time. Nevertheless, the overall measurement time can be reduced in a multichannel mode by applying the so-called "pipelined" method of signal acquisition. In this method, after a nuclei excitation cycle in one of the channels, the time required for this channel to restore the nuclear magnetization is used to excite the nuclei in the sensors of the other channels.

MEASUREMENT ACCURACY

The relative field measurements (or repeatability of measurements) have two main types of error:

(1) random error due to noise;

(2) error due to the instability of the synthesizer frequency.

Absolute measurements have, in addition to these errors, two more types of errors. The first is due to the uncertainty of the gyromagnetic ratio of nuclei taking into account the chemical shift and also due to the field distortions caused by themagnetic elements of the sensor, primarily the sample. The order of magnitude of this error is $(1–2)\cdot 10^{-6}$. The second error is related to the spatial uncertainty of the location of the measurement point, whose order of magnitude is about 10% of the field inhomogeneity in the sample volume.

The instability of the frequency synthesizer is caused by the instability of the reference generator, which does not exceed 10^{-6} in the temperature range from 10 to 50 50 °C. In the magnetometer, it is possible to use an external reference generator with a more stable frequency.

The random error caused by noise is characterized by its root-mean-square value at a measurement time of 1 s $(\sigma_F)_1$. The root-mean-square error $(\sigma_F)_1$ is determined by the width of the signal spectrum ΔF_s and the signal/noise ratio in the spectrum at an acquisition time of 1 s $(\mathrm{S/N})_1$ [6]:

$$(\sigma_F)_1 \cong \Delta F_\mathrm{s}/(\mathrm{S/N})_1. \tag{1}$$

The signal spectrum width ΔF_s can be represented as the sum of the intrinsic resonance width of the working substance ΔF_0 and the broadening due to the variation in the nuclear precession frequencies of the sample ΔF_n:

$$\Delta F_\mathrm{s} \cong \Delta F_0 + \Delta F_\mathrm{n} \cong 1/T_2 + \gamma \Delta B_\mathrm{n}/\pi, \tag{2}$$

where ΔB_n is the integral nonuniformity of the measured field in the sample.

The signal/noise ratio in the spectrum $(\mathrm{S/N})_1$ is determined by the parameters of the measured field, the sensor parameters, and the noise parameters of the input stages of the preamplifier [6]:

$$(\mathrm{S/N})_1 \equiv (\gamma\chi_0 B^2 V^{1/2} L^{1/2})/((\Delta F_\mathrm{s})^{1/2}(T_1)^{1/2}(S_\mathrm{n})^{1/2}), \tag{3}$$

where χ_0 is the nuclear magnetic susceptibility of the working substance, B is the induction of the measured field, V is the volume of the working substance (sample volume), L is the inductance of the coil sensor, T_1 is the transverse relaxation time of the working substance, and S_n is the spectral density of the electronics noise.

It follows from Eqs. (1)–(3) that the root-mean-square error $(\sigma_F)_1$ is largely determined by the parameters of the measured field; it decreases with increasing induction of the measured field and with decreasing field gradient at the measurement point.

TEST RESULTS

The tests were performed using a magnet with an induction of ∼0.15 T based on permanent magnets. The relative gradient of the field at the location of the sensor was $\sim(2\cdot 10^{-4}/\mathrm{cm})$. The test procedure consisted of continuous measurements of the field during a few minutes, followed by calculation of the standard deviation of the field from its mean value. As regular magnets have a significant temperature drift of the magnetic field, this effect of the drift on the measurements of the field $B(t)$ was eliminated by subtracting the linear component. Figure 4 shows the normalized measurement results after the procedure $(B - B_\mathrm{m})/B_\mathrm{m}$, where B_m is the mean value of the measured field.

The standard deviation σ of the measured field (or resolution) from the mean value was nearly $4\cdot 10^{-7}$ at a measurement time of ∼80 ms (no signal acquisition) and nearly $1.5\cdot 10^{-7}$ at a measurement time of ∼700 ms (number of acquisitions is $N_A = 8$). With increasing field, the magnetometer measurement accuracy generally increases, primarily due to an increase in the signal amplitude.

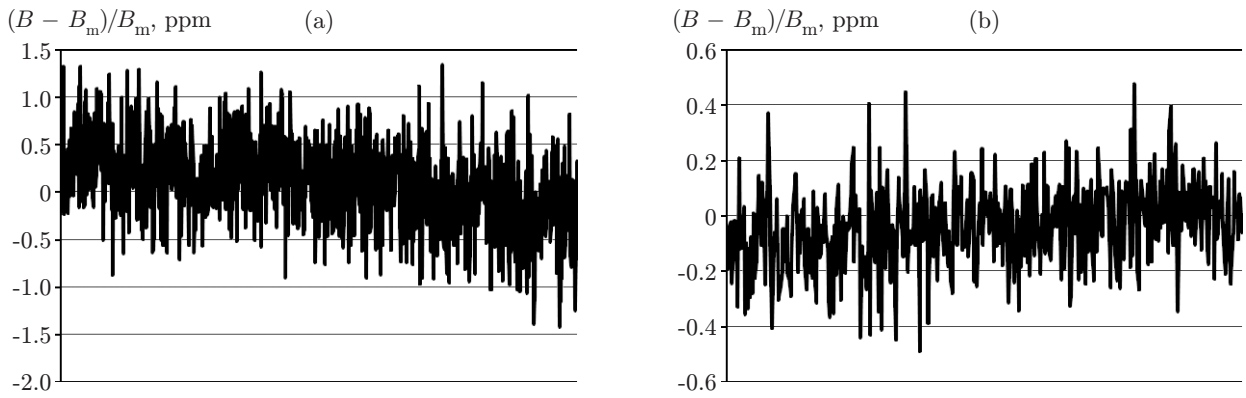

Fig. 4. Normalized measurement results of the magnetic field of the test magnet: (a) time of one measurement is ∼80 ms ($\sigma \approx 0.4$ ppm $= 4 \cdot 10^{-7}$); (b) time of one measurement is ∼700 ms ($\sigma \approx 0.15$ ppm $= 1.5 \cdot 10^{-7}$).

CONCLUSIONS

The proposed NMR magnetometer with one sensor measures uniform magnetic fields (with a relative gradient 2–3) $\cdot 10^{-4}$) with a relative accuracy of the order of 10^{-7} in the range from 0.05 to 2.5 T. This was achieved not only by optimization of the NMR sensors and minimization of electronics noise, but also by optimization of the signal processing. The digital spectrum analyzer included in the magnetometer automatically tunes its parameters to the NMR signal parameters, thereby providing signal filtering close to matched filtering and the maximum (for a given signal width) frequency resolution. Furthermore, despite the large amount of computations, the signal processing takes a relatively short time, (tens of milliseconds), and the total minimum time of one measurement is 70–80 ms. The pipelined method of signal acquisition used in the magnetometer allows measuring fields by four sensors almost simultaneously.

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