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Design improvements of nuclear magnetic resonance magnetometer to study magnetic mid-fields variations

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Abstract. The necessity to develop a magnetometer for variations research in the mid-fields magnetic strength with a relative error of 10^{-6} is justified. A modernization design of magnetometer to nuclear magnetic resonance with using to maser with flowing liquid is presented. The block diagram of nuclear magnetic resonance magnetometer is given. The core principle of its working is described. The results of experimental investigations of various variations to magnetic fields are presented. Further directions of modernization of the design of the magnetometer are determined.

Keywords: nuclear magnetic resonance, magnetic field, magnetometer, magnetic field variations, maser

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Материалы конференции
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Модернизация конструкции магнетометра на ядерном магнитном резонансе для исследования вариаций средних магнитных полей

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Аннотация. Обоснована необходимость разработки магнитометра для исследования вариаций средней напряженности магнитного поля с относительной погрешностью 10-6. Представлена конструкция модернизации магнитометра до ядерного магнитного резонанса с использованием мазера с проточной жидкостью. Приведена блок-схема магнитометра ядерного магнитного резонанса. Подробно представлен принцип его работы. Представлены результаты экспериментальных исследований различных вариаций магнитных полей. Определены дальнейшие направления модернизации конструкции магнитометра.

Ключевые слова: ядерный магнитный резонанс, магнитометр, магнитное поле, вариации магнитного поля, мазер

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Introduction

The development of technological progress and scientific investigations has led to an increase in the number of negative factors affecting various processes. [1–9]. A wide variety of negative factors led to the development of various instruments for measuring different kinds of physical parameters [10–19]. One of these parameters is the magnetic field [20–25], the amplitude and variation of which contains a lot of useful information [22–30]. That's why recently more and more areas of science and technologies have started demanding a lot of data on the magnetic field properties and characteristics [26–32]. Usually, it means that magnetic fields parameters must be measured and controlled with high accuracy. Such kind of problems can be successfully solved with modern devices like quantum magnetometers (which are based on nuclear magnetic resonance (NMR)). Them are one of most precise devices for measuring magnetic fields variations. [17, 18, 21, 30–33]. It should be noted that there are several designs to such nuclear-magnetic magnetometers. The very popular ones include devices based on a nuclear resonance filter [31]. In this device is realized to the frequency of the passive NMR line and the spin generator of phase or frequency self-tuning of the external generator frequency [32]. However, they do have disadvantages. For example, the first one has quite narrow NMR line, thus imposes grand demands for the tracking system. Because of it, several measurement problems occur. For example, temperature variations can cause noticeable significant dynamic errors. There are solutions to this issue, but they significantly complicate the overall device design.

Because of the reasons mentioned above, most of the industrial models of the quantum NMR magnetometers usually have upper limit around 100 μT. There are custom versions of these devices with upper limit of measurement before value of 0.0002 T, but they specifically designed to solve a small set of tasks. Overall, the values of the magnetic field were discussed above belong to so called low magnetic fields. However, the invention of generations of devices like particle accelerators, tokamaks, tracking devices for underwater objects requires new generations of magnetometers that will be able to measure the parameters of magnetic field in the diapason from 0.0002 to 0.2 mT with a relative error of measurement no more than 0.000001.

Nowadays, strength of the magnetic in in so called mid-fields (0.0002 T $< B <$ 1.0 T) is measured using the Hall effect magnetometers. However, even one of the best devices in this area (magnetic induction meter DX-180) has a relative error of measurement is the value of 0.000050.0005 in the measurement range from 0.01 mT to 300 mT with a resolution of 10100 nT. Therefore, it is extremely relevant to develop a new magnetometer design that will allow making measurements with a relative error no more than 0.000001.

Experiment and theory

Results of the analysis of current scientific state have shown that there is in fact only one manuscript that has been presented in the open sources and covers this topic [34]. This paper considers a scheme for self-tuning to the receiving circuit NMR resonant frequency of a maser with a flowing medium. Unfortunately, the data presented in [35] do not provide all required details, which prevents from full understanding of the device features. Also, it was found to be quite hard to reproduce the device design which is crucial to make required modifications to perform measuring of variations in mid-field magnetic strength in a wide range. This is vital for solving special tasks. Therefore, this article provides a detailed description of magnetometers components, as well as its structural diagram. The magnetometer is based on a maser with a flowing medium. In addition, the maser implements automatic tuning to the frequency of the NMR signal from the center of the generation zone.

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Fig. 1 shows the structure scheme of the magnetometer developed by us. In time a development of NMR magnetometer we used the data about the Benoit maser [35]. A mixture of alcohol and filtered tap water is used as a liquid.

Centrifugal pump 1 allows to vary consumption q from 0 to 0.020 l/s. The polarized vessel 3 is placed in magnet 2 (polarizer). Its magnet has an induction B_p = 1250 mT. The mixture magnetizes of magnet 2. Inverse population of the flowing liquid molecules is created by inverse coil 4 and radiofrequency generator 5. Not that inversion coil 4 is located in the magnet 3 stray field. Protons with the inverted (negative) magnetization are passed to the sensor 7 (for measuring magnetic field), which is located in electromagnet 6 that has an induction B_E (can take magnitude from 0 to 230 mT). The presented magnetometer uses 26 sensors. This allows us to measure the variations of magnetic field in the diapason from 0.2 to 200 mT.

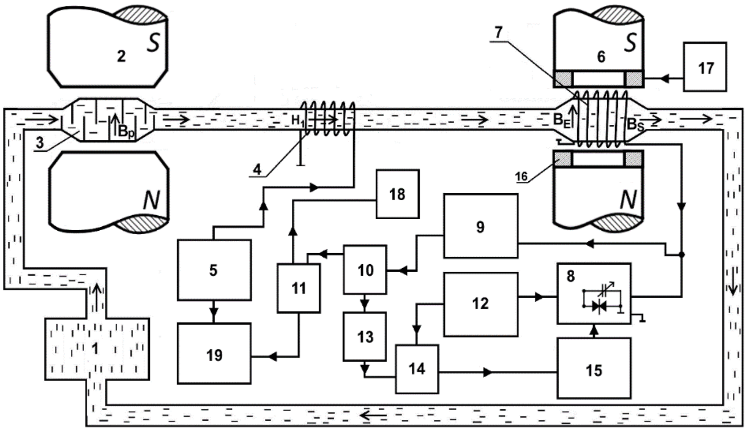

Fig. 1. Block diagram of a nuclear magnetic magnetometer: centrifugal pump *1*, polarizer magnet *2*, polarizer vessel *3*, pulse coil of water proton magnetization inversion *4*, radiofrequency generator *5*, electromagnet *6*, measuring sensor *7*, receiving circuit *8*, regenerator *9*, resonance amplifier *10*, registration device *11*, low-frequency generator *12*, low-frequency amplifier *13*, synchronous detector *14*, control circuit *15*, excitation coils *16*, pulse radiofrequency generator *17*, oscilloscope *18* and frequency meter *19*

Let us describe in detail how the magnetometer works with one of the sensors that allows measurements of field variations in the diapason 60 ± 13 mT. Measuring sensor 7 is a coil of 40 turns of copper wire with a winding length of 3 mm and a winding duty cycle η = 0.2. The receiving circuit intrinsic quality factor at frequency f_{nmr} = 2500 kHz of nuclear magnetic resonance has a value of more than 30. This circuit includes the coil of the measuring sensor 7 and is connected to the regenerator 9 input. In the developed by us of magnetometer design, it was decided to adjust level of positive feedback in the receiving circuit Q_c in order to compensate for the losses during the signal registration of nuclear magnetic resonance. To measure magnetic field variations in other sources, for example, 43 ± 14 mT, it is necessary to use another sensor 7.

This allowed to set Q_c set higher than the threshold quality factor of self-excitation that is equal to Q^{th}_c = 350. The generation EMF is created by magnetic moments of protons that are passed into the receiving circuit and then is amplified by resonance amplifier 10 with amplification bandwidth of 0.17 MHz and then is registered in the registration scheme 11. The frequency and the overall shape of the NMR signal is being monitored using an oscilloscope and a frequency meter that are connected to the registration scheme 11. Is should be noted that by changing the capacitance of the receiving system it is possible to reconFig. the maser generation zone. This is possible because it includes two oppositely connected variable capacitors (so called varicaps) that are connected to the audio generator 12 which provides an auxiliary voltage with a frequency of 10 to 20 Hz. The provided voltage basically modulates the circuit resonance frequency, which in turn modulates the voltage amplitude of the maser generation. Then, the lowfrequency amplifier 13 detects the value of the modulated signal which comes from the resonant amplifier 10. Then the signal is passed to the synchronous detector 14. Note that this detector also has an input that receives the voltage, which is the reference, created by the generator 12. If there is a detuning between the maser generation frequency and the resonant frequency of the receiving circuit, then at the output of the synchronous detector 13 a control voltage appears. This voltage is of interest because its sign and amplitude are proportional to the detuning. That's why it is fed to the control scheme 15 which was developed and assembled using integrated operational amplifiers. Finally, this signal is passed to varicaps that allows to control the receiving circuit resonant frequency thus adjusting it to the resonant line center.

The transverse relaxation time T_2 can be evaluated in two ways:

by exciting the spin system with a $\pi/2$ pulse and measuring the free induction signal decay time;

by modulating the magnetic field while disconnecting generator 5 from nutation coils 4 and switching the regenerator to self-excitation mode, and measuring the NMR signal decay time.

Having the relaxation time value determined, variations of the magnetic field strength can be evaluated as follows:

$$\Delta\omega_{\max} = \frac{1}{\left(T_2\sqrt{Q_c / Q_0} - 1\right)}, \tag{1}$$

where Q_c is the receiving circuit quality factor and Q_0 is the threshold quality factor at zero detuning.

The measurement error of the magnetic field in the developed magnetometer is determined as follows. Measurements of the induction B_0 are carried out at the resonant frequency of protons (frequency measurements).

$$\gamma B_0 = \omega_0. \tag{2}$$

The frequency value ω_0 is determined using a modern frequency comparator (for example, on a gas rubidium cell or a cesium atomic beam tube) with an accuracy of 10^{-9}. The gyromagnetic ratio γ for protons is determined with an accuracy of 10^{-7}. According to the rules of indirect measurements, the B_0 measurement error will be no worse than 10^{-6}. The signal-to-noise ratio during measurements should be greater than 3.5 for direct measurements or greater than 1.5 for accumulation measurements. In the developed magnetometer, the signal-to-noise ratio is greater than 5. If necessary, it can be increased at least 2–3 times by increasing the induction B_p of the polarizer magnet. The higher the magnetization of the flowing liquid, the higher the signal-to-noise ratio of the registered NMR signal.

To obtain a higher accuracy for all NMR magnetometers operating at the resonant frequency of protons, it is necessary to determine the value of γ with an error of 10^{-8} or less.

Experimental results and discussion

An example of the NMR signal registered with using the modulation of the magnetic field B_E = 59.52 mT is shown in Fig. 2. The envelope decay of the signal peaks is used to determine the transverse relaxation effective time T^*_2 of the media (in this case, it is a flowing liquid). Specifically, T^*_2 is measuring by the decrease in NMR signal amplitude in range from U_{max} to $U_t = U_{max}/10$.

Knowing the effective relaxation time, it is possible to determine the relaxation time T_2 as follows:

$$\frac{1}{T_2^*} = \frac{1}{T_2} + \frac{\gamma * \Delta H}{\pi}, \tag{3}$$

where ΔH is the magnetic field uniformity in the in the place of the sensor 7.

The magnetic field uniformity is roughly equal to 10^{-4} cm^{-1} in the magnetometer design considered in this paper.

The magnetization M_0 of a liquid entering the sensor 7 is one of the important characteristics for performing measurements. That is because the amplitude of the recorded NMR signal is proportional to the value of magnetization $\mathbf{M}_0$. Therefore, the higher the $\mathbf{M}_0$ value, the higher the measurement accuracy of the magnetic field variation. Thus, it is feasible to conduct research on the correlation between flow rate q and the NMR signal amplitude U using the considered sensor 7. Results of this research are shown in Fig. 3.

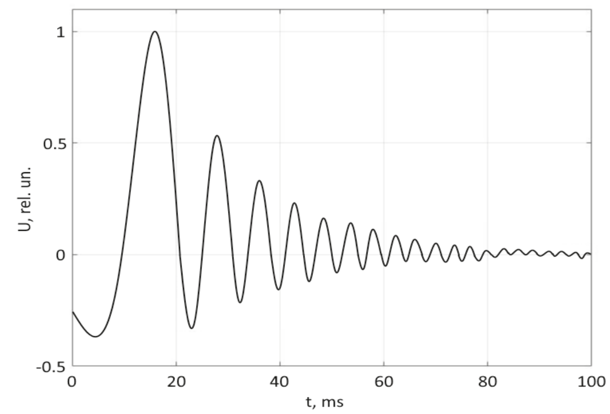

Fig. 2. Registered NMR signal from a mixture of alcohol with filtered tap water at $T = 295.3$ K

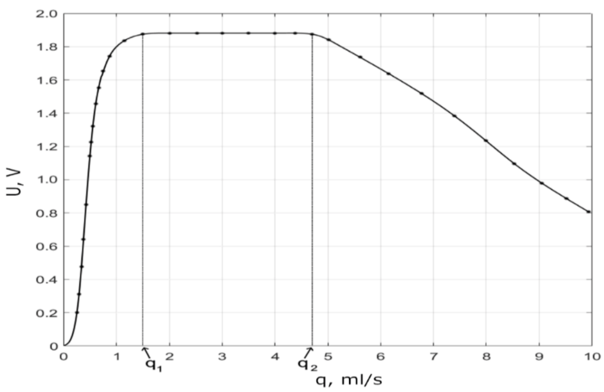

Fig. 3. Dependence of the NMR signal amplitude U on the flow rate q of a mixture of alcohol with a filtered tap water at $T = 295.3$ K

According to the results of the research, there is a fairly wide range of a flow rate q in which the signal-to-noise (SNR) ratio is high enough (i.e., more than 20). Specifically, this range is from $q_1 = 0.02 \pm 1.59$ ml/s to $q_2 = 0.05 \pm 4.74$ ml/s. If the device operates in this range, significant changes in the flow rate (up to 10 %) will not affect the measurement error if the overall flowrate is close to the middle of the range.

Using the obtained data, the transverse relaxation time T_2 was determined in two described above ways for fluid flow $q = 0.02 \pm 2.99$ ml/s. The result of the free induction decay method is $T_2 = 0.18 \pm 23.21$ ms. The result of the free magnetic field modulation is $T_2 = 0.23 \pm 23.04$ ms. It should be noted that in order to estimate a measurement error using standard methods, in both cases measurements were repeated 10 times with subsequent data averaging.

Also, to additionally validate the obtained data, characteristics of the same solution at the temperature $T = 294.9$ K were measured on a stationary NMR relaxometer Minispec mq 20M (made by BRUKER company). The measured value was $T_2 = 0.065 \pm 23.198$ ms. Thus, it can be concluded that all obtained transverse relaxation time values coincided within the measurement error.

Conclusion

The results of research have showed that the self-generating NMR magnetometer considered in this paper allows measuring variations of the magnetic field in the diapason of 0.2 to 200 mT. This becomes possible because magnetometer uses automatic tuning to center of the generation zone for the proton resonance frequency. What's more important, in both dynamic and static modes the relative measurement error doesn't exceed 10^{-6} at input signal variations with frequency $f_v \leq 0.25$ Hz and amplitude up to from $^{5-}10$ to $^{2-}10$ T, depending on the measuring range.

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