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A portable digital multipulse NQR spectrometer for the study of the sensory properties, structure and defects in layered semiconductors

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***Abstract*—The structure of a digital pulse coherent NQR spectrometer with a minimum number of functional units for the investigation of GaSe and InSe layered semiconductor crystals is proposed. The feature of the proposed measuring setup is the implementation of all algorithms of digital processing and signal synthesis in the basis of one statically-configured FPGA EP4CE15E22C8. The digital 48-bit frequency synthesizer provides the formation of carrier frequency oscillations, which fill of the excitation pulses in the NQR frequencies range 1 – 50 MHz (isotopes ^{14}N, ^{69}Ga, ^{71}Ga, ^{113}In, ^{115}In, and others). Reliable gating of the receiver in the pauses between the excitation pulses (up to 100 dB for the carrier frequency) is ensured by a circuit diagram of three stages of the same type on double gate field-effect transistors.**

***Keywords*—radio spectrometer; NQR; transient process; FPGA**

I. INTRODUCTION

The method of nuclear quadrupole resonance (NQR) is based on the absorption of high-frequency energy by changing the orientation of the quadrupole moments of atomic nuclei in the heterogeneous electric field created by external charges relative to the nucleus. The levels of quadrupole energy in a solid substance arise when quadrupole moments interact with a heterogeneous electric field at the location of the resonating nucleus, therefore the NQR spectrum reflects the distribution of electron density near a certain atom. This is the uniqueness of the NQR method in the study of the fine features of the structure of chemical compounds [1].

The presence of the quadrupole moments of the gallium and indium isotopes in GaSe and InSe allows using NQR spectroscopy to study the crystal structure of these crystalline compounds [1, 2]. This method is extremely sensitive to changes in the local electric field in the crystal lattice, which can be caused even by slight shifts in the crystal matrix [3]. The latter may be due to the movement of dislocations, rearrangement in the system of point and structural defects, deformation, and machining of crystals. The advantage of NQR over other integral methods is due to the local nature of the information obtained, the non-destructive effects and high resolution of the resonance spectra, which is provided even at room temperatures.

When registering NQR by pulse methods, there is a problem in eliminating the overload of the radio spectrometer receiver during the action of the excitation pulse and in suppressing free oscillations after its termination [4]. The latter is due to the residual transition process in the oscillating circuit and can significantly increase the "dead" zone of reception of the free induction decay signal (FID) or even completely prevent its reception and amplification. In this case, useful information is lost, which is important when applying the Fourier transform of the spin induction signal to display the NQR resonance spectrum. Methods that make possible the attenuation of the transient process were considered in [2, 5]. In particular, in [2], a key device for single gate field-effect transistors was proposed, which provides carrier suppression ratio during the action of high-power excitation pulses by only 60 dB. A similar solution for a key device made on a bipolar transistor is effectively used in a pulse spectrometer when detecting NQR signals of ^{14}N isotope in the low-frequency range (0.5 – 5 MHz) [5]. However, in this case there is a decrease in the carrier suppression ratio at an increase in its frequency to 20 – 30 MHz.

This paper proposes the structure of NQR digital pulse radio spectrometer, as well as the method for attenuation of transient processes in its input circuit and receiver.

II. BLOCK DIAGRAM OF A PORTABLE NQR SPECTROMETER

The pulse method for detecting nuclear quadrupole resonance signals requires the use of high-power radio-frequency excitation pulses and high-sensitivity receiving equipment [6]. A block diagram of a laboratory coherent radio spectrometer, proposed for pulse observation of NQR in GaSe and InSe semiconductors, is shown in Fig. 1.

The feature of the proposed radio spectrometer is its implementation on the basis of a multifunctional software-controlled digital computational core. Based on the syntax for modeling dynamic modes of logical structures, simulation models and software algorithms have been developed for implementing the basic functional units of the NQR pulse spectrometer in a single module based on field-programmable gate array (FPGA) Intel (Altera) Cyclone IV EP4CE15E22C8.

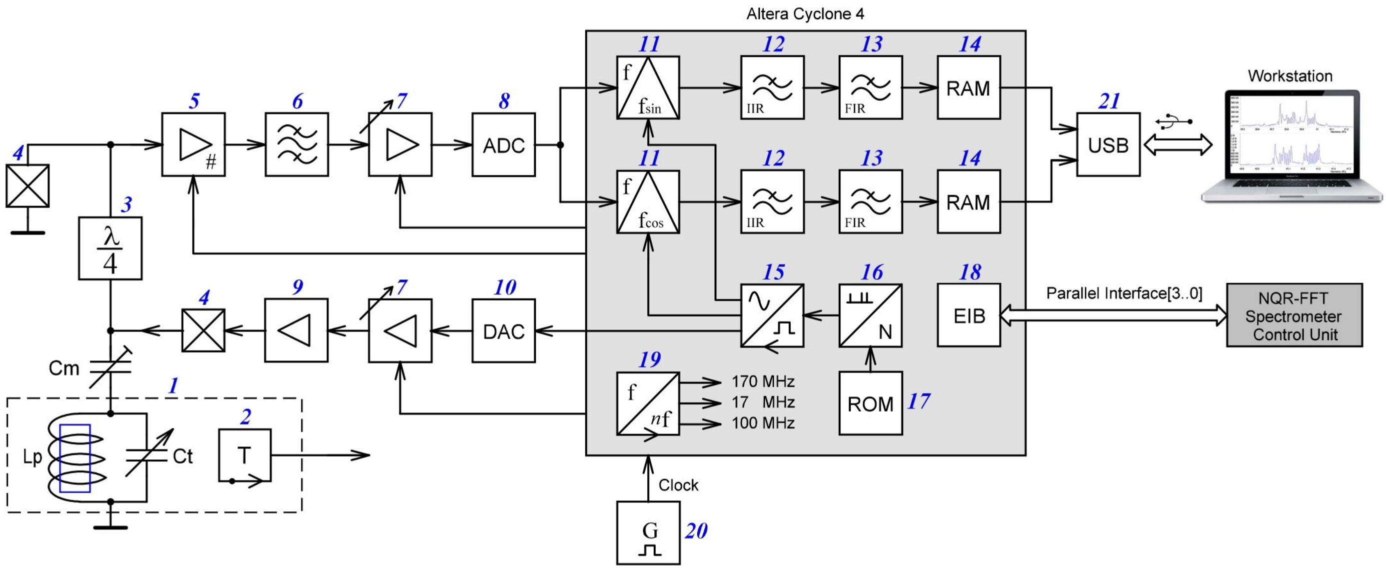

Fig. 1. Block diagram of portable digital multipulse NQR spectrometer: 1 – measuring cell; 2 – temperature sensor in the cell; 3 – λ/4-cable; 4 – diode limiters; 5 – gated amplifier; 6 – bandpass filter; 7 – code-controlled amplifiers; 8 – ADC; 9 – output power amplifier; 10 – DAC; 11 – digital multipliers; 12 – IIR type digital filters; 13 – FIR type digital filters; 14 – RAM; 15 – three-channel DDS; 16 – pulse sequence programmer; 17 – ROM; 18 – spectrometer interface bus controller; 19 – multi-channel PLL; 20 – quartz clock generator; 21 – USB interface controller; Cm – matching capacitor; Ct – tuning capacitor; Lp – probe coil.

The source of the carrier frequency is a three-channel Direct Digital Synthesizer (DDS) (***15***) based on a 48-bit phase battery with the possibility of high-speed frequency and phase shift keying. It provides the generation of NQR excitation pulses in the frequency range of 1 – 50 MHz. The step of setting the frequency of carrier oscillations is $\Delta f_{out} \approx 1\times10^{-6}$ Hz. The pulse sequence programmer (***16***) provides the formation of 90º-degree excitation pulses with a duration of 0.1 – 20 µs and a minimum repetition period of 0.1 µs. The duration of the pause between the excitation pulses is adjustable in the range of 0.1 µs – 1 s. Other time lengths, for example, in the Carr-Purcell sequence, the length of 180-degree pulses and pauses between them is set automatically according to the selected program recorded in the non-volatile memory (***17***). The frequency of the carrier wave, the duration of a 90º-degree excitation pulse, the duration of the pause between pulses and the type of sequence come from the spectrometer control unit to the digital computational core through a four-bit parallel interface.

The analog path of the radio spectrometer is implemented as a functionally complete transmitter-receiver unit containing a gated amplifier (***5***), a band-pass filter (***6***), and code-controlled amplifiers (***7***). The output broadband power amplifier (***9***) is a high-frequency transmitter loaded on an LC circuit (***1***), in the coil of which the test substance is located. The transmitter allows one to develop powerful *δ*-shaped pulses in the coil with an initial pulse power of 1 kW in the frequency range of 1 – 50 MHz.

The radio spectrometer receiver was developed with Software Defined Radio (SDR) technology using the Digital Down-Converter (DDC) principle, which significantly reduced the number of analog path stages and, therefore, significantly reduced the noise contamination of the FID signal and the asymmetry of the detected signal parameters. In the experimental model of the spectrometer, a high-speed 12-bit analog-to-digital converter (ADC) AD9230BCPZ (170 MSPS conversion rate) was used to digitize the response signal in the resonance frequency range of 1 – 50 MHz. Quadrature signals of the reference frequency enter the inputs of the multipliers (***11***), where their multiplication with the information signal take place. Since ADC data and DDS reference signals are 12-bit, we get a 24-bit number as a result of multiplication. The multiplication operation will lead to the transfer of the signal spectrum to the low frequency range. In this case, further work with the digital signal sampled at 170 million samples per second, is no longer appropriate and resampling to a lower frequency should be made. Before this operation, a low pass filter (LPF) is required. In the SDR structure, a series connection of IIR-filters (***12***) is applied, which provide a reduction of the sampling rate by 10 and compensation FIR-filters (***13***), which provide to equalize the frequency response of the receiver.

A data acquisition system based on a two-channel bidirectional USB interface FT2232H (***21***) for processing the response signals of a nuclear spin system with the subsequent allocation of the NQR spectrum is used. The LabVIEW software for Fast Fourier Transform and averaging FID signals is developed. The interval between measurements is set by the frequency of the starting pulses, which are synchronized with the USB interface.

The number of turns of the probe coil (Lp) depends on the operating frequency range and the sample volume. For GaSe and InSe ingots grown by the Bridgman method, the coil diameter can be 18 – 20 mm and have 8 turns of silver-plated copper wire (for 20 MHz frequency) or 5 turns for 40 MHz frequency.

III. CIRCUIT DIAGRAM OF A GATED AMPLIFIER

The purpose of the input part of the spectrometer is to send a powerful radio pulse with high-frequency filling to the sample coil for a very short time (1 μs – 20 μs) and immediately proceed to receive the signal of spin induction of quadrupolar nuclei. The problem is to eliminate the overload of the receiver during the action of the excitation pulse and suppress free oscillations after its termination. The latter is due to the residual transient process in the oscillating circuit and can significantly increase the "dead" zone of receiving the induction signal or even completely eliminate its reception and amplification. To attenuate the transient process, a circuit diagram of a 3-stage gated preamplifier of the receiver of an NQR spectrometer, built on pairs of field-effect transistors with insulated gates and low intrinsic noise coefficient, was proposed (Fig. 2).

Consider the work of a separate circuit on Q1 and Q2. Limited by the diodes D1, D2 to the level of 0.4 – 0.6 V, the residual oscillations of the carrier frequency during the effect of the gating pulse through isolation capacitor C1 are fed to the first gate of transistor Q1. From the inverter output on the 74LVC1G4 chip, a logical "zero" is applied to the second gate of the Q1 transistor, which gates the transistor and thereby suppresses residual oscillations of the carrier frequency. In parallel, the transistor Q2 is open to the passage of the constant component by supplying a non-inverted gating pulse (logical "1") to the second gate. At the end of the gating pulse functions of the transistors Q1 and Q2 are swapped. At the time of the absence of a gating pulse, a logical "zero" enters the second gate of the transistor Q2 and the transistor gates. At the same time, the Q1 transistor opens due to the logic "1" supply to the second gate and passes the constant component and the signal through the load - the winding of the isolation transformer T1. The maximum switching time of the 74LVC1G04 logic element is no more than 1.6 ns and, therefore, switching on the excitation pulse and transfer to the induction signal reception mode does not lead to a noticeable interruption of the constant component in the drain of the field-effect transistors and in the primary winding of the isolation transformer. The following cascades of the same type on transistors Q3 – Q6 work in a similar way and enhance the effect of suppressing the residual signal of the carrier frequency and the transient process arising from the switching of pulses.

IV. THE PRINCIPLE OF CONSTRUCTION AND EXPERIMENTAL STUDIES OF THE RECEIVER OF THE NQR SPECTROMETER

A. The design of the gated amplifier

According to the results of circuit diagram simulation, the receiver of the NQR pulse spectrometer was developed. The analog part of the receiver, in addition to the spin-induction preamplifier under consideration, also contains a bandpass filter and an RF matching amplifier. The choice of transistors BF998 is due to the low coefficient of intrinsic noise (about 0.6 dB at a frequency of 200 MHz). The mode of operation of transistors Q1 – Q6 is set by adjusting the bias voltage +Ub. In addition, the maximum sensitivity of the device was observed at +Ub = 0.3 V. The matching transformers T1 – T3 were made on Amidon ferrite cores BN-43-2402, which are designed for an optimal frequency range of 5 – 400 MHz. The ratio of turns of the windings is 1:2. The bandpass filter is introduced to attenuate excess noise and spurious spectral components. The bandwidth of this filter is set between 0.2 – 1 MHz and can be tuned in frequency. The choice of this band is mainly due to the width of the resonance signal spectrum. For the NQR spectra of gallium and indium, the optimum bandwidth of this filter is 0.3 – 0.5 MHz in the frequency range of the spin transitions 19 – 41 MHz. A matching amplifier is needed to match the output resistance of the filter with the wave resistance of the connecting cables (50 Ω) and, accordingly, with the input impedance of the code-controlled amplifier (Fig. 1). The basis for the matching amplifier is LT6201 ultra-low noise high-frequency operating amplifiers with a bandwidth of 165 MHz. The receiver is located in a metal case with functional elements separated by shielding partitions. To prevent the occurrence of spurious oscillations and the simultaneous reduction of noise and interference, a system of isolating and blocking links was used. The device is powered by a modular linear multichannel voltage stabilizer based on LD1117, LM317 and LM337 voltage stabilizers.

The proposed receiver based on a gated amplifier with a transformer coupling between the stages, can also be used to observe NQR in the low frequency range (^{14}N isotope and others). To do this, it is necessary to calculate the corresponding parameters of the elements of the input circuit (Cm, Ct, Lp) and use isolation transformers of a different configuration.

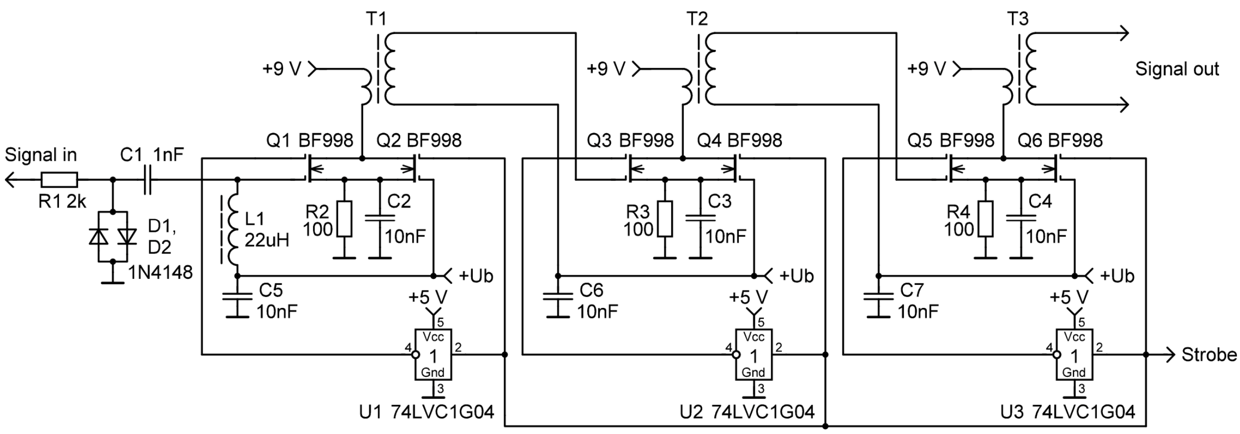

Fig. 2. Preamplifier of spin induction signal with a transformer coupling between the stages.

B. *Research results and discussion*

The research on the laboratory layout of the proposed receiver based on a gated amplifier with a transformer coupling between the stages was carried out in conjunction with a digital multipulse coherent NQR radio spectrometer.

As can be seen from Fig. 3, the carrier suppression ratio varies with its value in the range from 73 dB to almost 100 dB. The total voltage gain of the analog part of the receiver is about 50 dB in the frequency range of 6 – 30 MHz, the unevenness of the frequency response is ±2 dB (Fig. 4). The gain reduction at frequencies below 5 MHz is due to the properties of the used ferrite cores. The smooth decay of the frequency response at frequencies above 30 MHz is explained by the frequency dependence of the impedance of the windings of isolation interstage transformers. Obviously, an optimal frequency range has been obtained for a given circuit, since as the inductances of the windings decrease, an upward shift in the lower limit of the frequency characteristic is observed, and as they increase, the amplifier loses its broadband and acquires resonance properties.

The proposed measuring setup (Fig. 5), in particular the receiver based on a gated amplifier with a transformer coupling between the stages, can also be used to observe NQR in the low frequency range (^{14}N isotope and others).

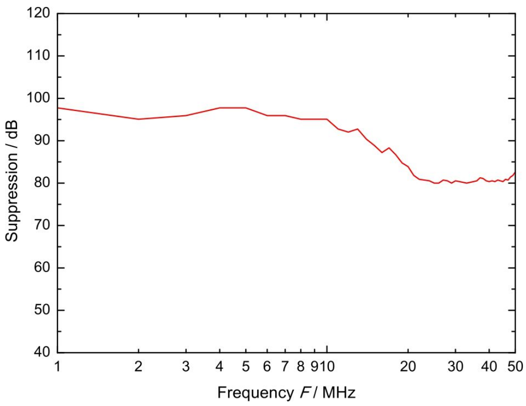

Fig. 3. Frequency dependence of the carrier suppression ratio for the proposed receiver of NQR measuring setup.

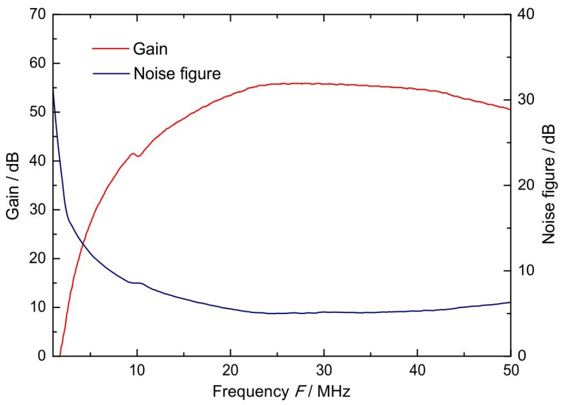

Fig. 4. Gain and noise figure versus frequency of the proposed receiver of the NQR measuring setup.

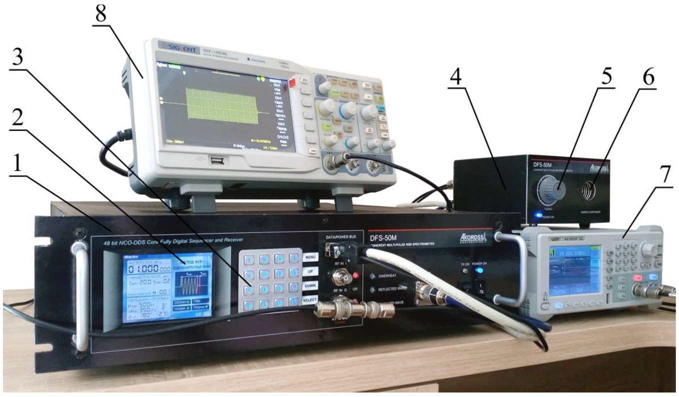

Fig. 5. Measurement setup for the observation of NQR: 1 – portable digital multipulse NQR spectrometer, 2 – graphical display, 3 – settings panel, 4 – NQR signal sensor, 5 – tuning capacitor, 6 – probe coil, 7 – signal generator, 8 – oscilloscope.

CONCLUSIONS

A block diagram of a digital coherent radio spectrometer for studying GaSe and InSe layered semiconductors by the NQR method is presented. The device is differ by the minimum number of functional units while maintaining the sensitivity required for registration of the NQR spin induction signals. Circuit design solutions are proposed to eliminate the "ringing" of the receiving coil of the oscillating circuit and to suppress the transient process in the receiver of the NQR pulse radio spectrometer. Reliable gating of the receiver in the pauses between the excitation pulses (up to 100 dB for the carrier frequency) is ensured by 3 stages of the same type on double gate field-effect transistors.

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