

PULSE SEQUENCE SHAPER FOR RADIOSPECTROSCOPY AND RELAXATION METHODS IN NQR

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Abstract

A pulse sequence shaper for the pursuance of the research using a wide spectrum of radiospectroscopy and relaxation methods in NQR is proposed. The distinctive feature of this product is its implementation with the application of a multi-functional programmable frequency synthesizer suitable for high-speed amplitude and phase manipulations.

Keywords: NQR, pulse sequence, Field – Programmable Gate Array, frequency synthesizer.

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1. Introduction

The use of relaxation spectroscopy methods is currently an important tool for studying the structure, defects and dynamic characteristics of crystal lattice of semiconductor structures by means of nuclear quadruple resonance (NQR) [1–3].

Pulsed methods of NQR observation are widely used for solving various problems of solid state physics. As compared to stationary methods, pulsed methods offer an important advantage, namely the possibility of fast and precise measurement of relaxation times. In the case of a pulsed method for investigation of the NQR spectral characteristics, additional information follows from the measured values of spin-spin and spin-lattice relaxation times, that result from the interaction inside the spin system and alternative degrees of freedom inside the lattice. These parameters characterize the dynamics of lattice and spins [4].

One of the functional units of an NQR Fourier spectrometer is a pulse sequence shaper. It programs the work of pulse keys that assure shaping of NQR excitation pulses. The programmer should form specified pulse sequences with the pulse length and the intervals between pulses regulated with pre-assigned accuracy. If the programmer forms a series of pulses and consecutively rotates the system of nuclear spins, a series of gradually attenuated signals occurs. In several tenths of pulses the signals may even not occur. This is caused by the incoherence of spin rotation or “phase memory loss” due to different microsystem interactions [5].

The majority of excitation pulse shapers for nuclear resonance and relaxation studies proposed by developers have a number of disadvantages. In particular, most of them are made as PC expansion cards and require development of a dedicated software which imposes

limitations on their mobility [6]. Moreover, by virtue of their wide functionality and, accordingly, high cost, such devices are not affordable for the majority of research laboratories.

This paper presents implementation of a pulse sequence shaper for radio-spectroscopy and relaxation methods in NQR. The use of Field – Programmable Gate Array (FPGA) assured shaping of NQR excitation pulses with preassigned accuracy and possible change in the configuration of sequence parameters [7].

2. Rationale of choosing the method

The existing pulsed spectroscopy and relaxometry methods of NQR are conventionally divided into incoherent and coherent systems that are mainly noted for the presence of phase synchronization of excitation pulses with carrier frequency oscillations (Fig. 1). The elaborated oscillation packages of carrier frequency (Fig. 1*d*) are probing pulses whose leading edges are defined by trigger pulses (Fig. 1*b*) and are tied to the oscillation phase of carrier frequency. In this way the coherence of spectrometer operation is provided.

For performing the relaxation studies both coherence of shaped pulses and the accuracy of determination of 90°-th and 180°-th pulses duration and pauses in the sequence are important [4].

Since the NQR signal frequency for a certain compound is a function of the electric field gradient value, the exact value of resonance frequency is unknown. That is, the basic frequency must be varied and, accordingly, the duration of 90°-th and 180°-th pulses must be selected on a case-by-case basis. It is difficult to be achieved using the methods considered in [8], as phase binding of probing pulses to carrier oscillation causes the time error which is accumulated with increasing the number of pulses in the sequence (Fig. 1*d*).

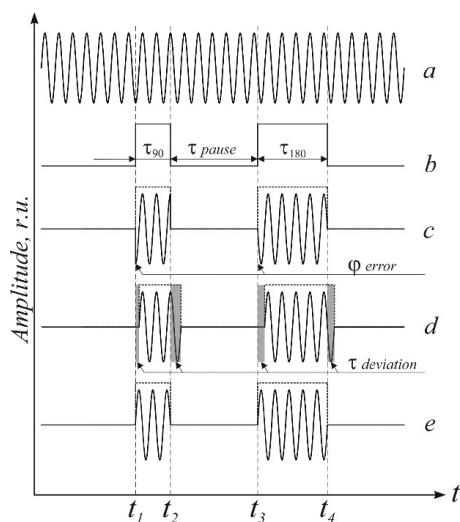


Fig. 1. The time diagrams of shaping NQR excitation pulses: *a*-carrier frequency oscillations; *b*-the probing pulse envelope; the probing pulse at – incoherent shaping-*c*; coherent shaping-*d*; use of a controlled synthesizer-*e*.

The range of application of NQR methods requires the use of shapers with the possibility of setting and changing many pulse parameters in the sequences, which complicates implementation and increases the cost of NQR equipment. The advent of reconfigurable FPGAs facilitates this task and makes possible implementation of pulsed methods of NQR on their basis, since the use of FPGA allows to create not only carrier oscillations with unvarying

frequency and initial phase, but also a flexible system of shaping coherent excitation pulses of NQR signals.

3. Algorithm design for FPGA

The pulse sequence shaper is a hardware-software solution. The program algorithm was designed by methods of simulation and development of very large-scale integrated circuits and systems on a chip by Altera Corporation [9]. The pulse sequence shaping diagram on the basis of FPGA is depicted in Fig. 2.

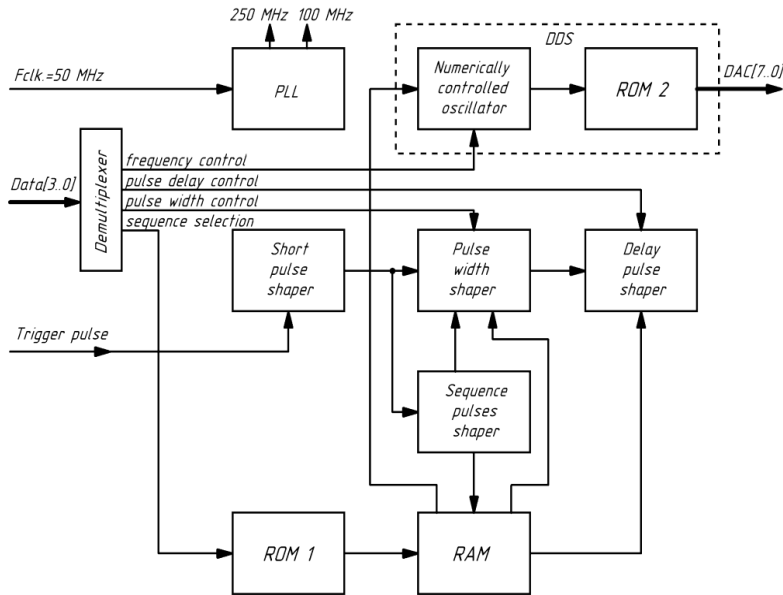


Fig. 2. The pulse sequence shaping diagram based on a controlled synthesizer.

The distinguishing feature of this product is its implementation with the use of a multi-functional program-controlled frequency synthesizer with the possibility of fast frequency and phase manipulations (Fig. 3). Formation of carrier high-frequency oscillations is based on Direct Digital Synthesizers (or DDS) that are widely applied in radio-electronic devices and telecommunication systems [10]. The synthesizer is based on a 48-bit phase accumulator “altaccumulate0” which forms the code sequence of signal instantaneous phase that varies linearly. The modules “phase_mux”, “phase_adder” and “phase_reg” serve to implement the phase manipulation of the carrier frequency of DDS output signal.

The PLL (Phase Locked Loop) module is a programmable system that generates oscillations with the frequencies $f_{clk1} = 250$ MHz and $f_{clk2} = 100$ MHz with an external clock generator operating at the frequency of 50 MHz (Fig. 2). The frequency f_{clk1} is used as the reference frequency for the digital frequency synthesizer, whereas f_{clk2} is used for operation of the pulse former (Fig. 4) with the minimum pulse duration $\tau = 100$ nsec. Readings of the frequency synthesizer output signal are written in the table “DDS_rom” (Fig. 3).

The pulse sequence shaping circuit in the program editor ALTERA Quartus Web Edition corresponding to the designed algorithm is presented in Fig. 4.

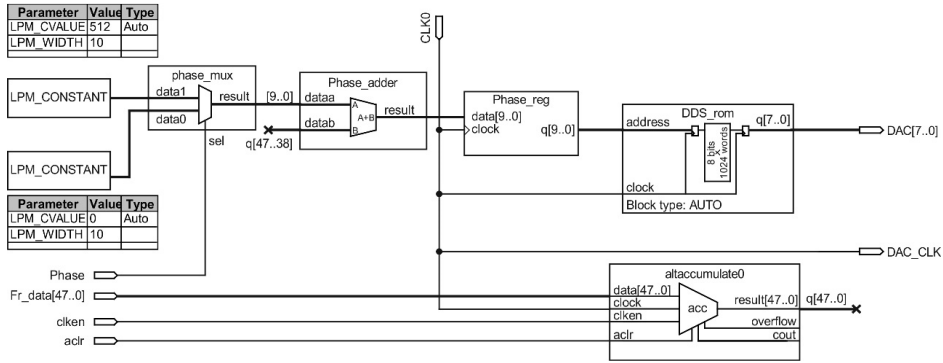


Fig. 3. The Direct Digital Synthesizer.

The multiplier “mult 1” enables introduction of the input frequency code immediately in the form of a numerical constant corresponding to the anticipated numerical value of generated frequency. It multiplies the input code of frequency K_1 by the constant $K_2 = 11258999$ that sets the code $K = K_1 K_2$ of the synthesizer frequency step $\Delta f = 10$ Hz.

Dependence of the signal frequency f_{out} at the output of numerically controlled generator on the reference clock frequency f_{clk} , the capacity of phase accumulator M and the frequency code K is determined as follows [11]:

$$f_{out} = \frac{K \times f_{clk}}{2^M}. \quad (1)$$

In so doing, the frequency step does not depend on its value and is equal to:

$$\Delta f_{out} = \frac{f_{clk}}{2^M}. \quad (2)$$

In our case the operating frequency of phase accumulator with the capacity $M = 48$ bits is equal to $f_{clk1} = 250$ MHz. Thus, the frequency step will be $\Delta f_{out} \approx 1 \times 10^{-6}$ Hz. So, to shape a signal with the frequency f_{out} , to the synthesizer input “Fr_data[47..0]” one should feed a 24-bit frequency code determined by the following expression:

$$K_1 = \frac{2^M f_{out}}{f_{clk} K_2} = 25 \times 10^6 \frac{f_{out}}{f_{clk}}. \quad (3)$$

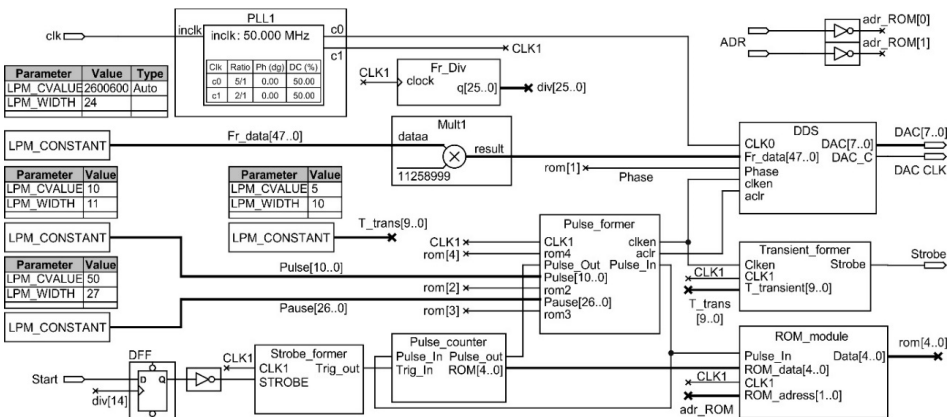


Fig. 4. Implementation of the pulse sequence shaping circuit in ALTERA environment Quartus Web Edition.

The sequence generation starts synchronously with the input gating pulse. The data on the frequency of carrier oscillation, duration of 90°-th pulse, pulse spacing and sequence type come from the external control unit along the Data bus [3..0] (Fig. 2). The duration of 90°-th pulse is set in the range of 0.1÷20 µsec with the step of 0.1 µsec. Pulse spacing is controlled in the range of 0.1 µsec÷1 sec. All the other time intervals, e.g. in the Carr-Purcell sequence [4], the duration of 180°-th pulses and their spacing are set automatically, according to a selected program recorded in ROM 1. The number of pulses in a series is assigned within 1÷30. Information on the parameters of this or another sequence through buffered RAM is passed to a series of actuation devices, namely the program-controlled generator, and the pulse and pause duration formers.

The structure of file fragment written in ROM 1 (Fig. 5) shows arrangement of information bits in the words. The first bit is responsible for the presence of pulse in the sequence. Next bits control the pulse duration, the pause duration, and enable the phase shift. These bits define the rules of the 90°-th and 180°-th pulses. If bits 1 and 2 are set to “0”, the durations of pulses and pauses are set by the external control unit. If bits 1 and 2 are set to “1”, the durations of pulses and pauses are twice longer.

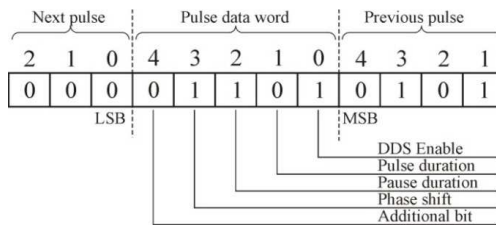


Fig. 5. Memory write structure ROM 1.

Simulation of the designed algorithm operation was done by means of Simulator Tool from ALTERA. The simulation results in Fig. 6 show that the values of basic frequency, initial phases, pulse duration and pause duration in a sequence correspond to those prerecorded in ROM 1. The volume of used FPGA EP1C6T144C8N hardware resources was the following: total of logic elements – 1435 (24%), total of memory bits – 50112 (54%), total of PLL – 1 (50%).



Fig. 6. Simulation of device operation: 1 – 50 MHz clock pulses; 2 – start pulse; 3 – sync pulse; 4 – 250 MHz clock pulses; 5 – memory address bus; 6 – memory address bus; 7 – DDS synthesizer enable pulses; 8 – data bus on DAC; 9 – simulated analog signal; 10 – synchronous detector control pulses; 11 – gating pulses.

The designed algorithm also provides shaping the reference signal for operation of the external synchronous detector (Fig. 6, 10) and the gating pulses for control of the radio spectrometer reception path (Fig. 6, 11). The work of the shaper allows for the possibility of setting additional time at the end of each shaped probing pulse for the suppression of receiving coil “ringing” after the action of probing excitation pulse [12].

4. Research results

For firmware implementation of the pulse sequence shaper a system board of pulsed NQR-spectrometer was developed. It consists of digital-to-analog and analog-to-digital converters, a low-pass filter, matching and buffer amplifiers, configuration and supply circuits, and other functional elements (Fig. 7). The product proposed here is based on FPGA EP1C6T144C8N of Altera Cyclone family [13]. The internal structure of this FPGA has almost 6000 logical elements that can be used to implement digital functional units due to the syntax of simulation of the dynamic modes of logical structures. The specific feature of FPGA employed is the presence of two functionally independent phase lock modules.

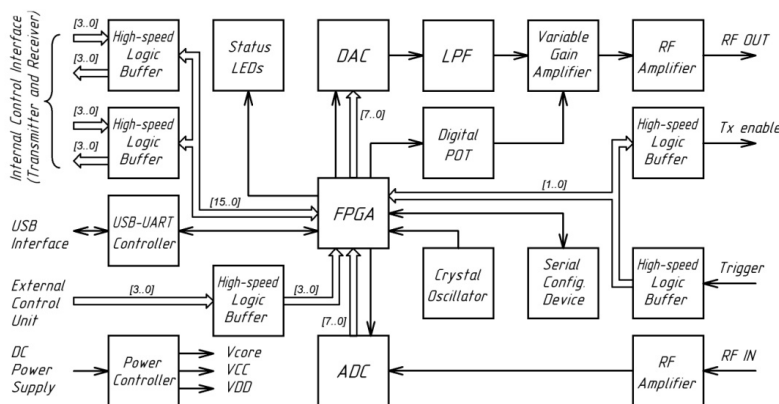


Fig. 7. A block diagram of the spectrometer system board.

The FPGA microcircuit with a configuration memory and a crystal oscillator forms the system “core” which is installed on the system board and, if necessary, can be replaced by a similar one with a higher operational speed.

The non-volatile memory of FPGA has information recorded on 30 various types of sequences, use of which assures implementation of a wide range of radio-spectroscopy and relaxation methods in NQR, in particular, for determination of relaxation times T_1 and T_2 (CPMG-type sequence), provision of accumulation of weak NQR signals (SSFP and SLSE sequences) with fast and low relaxations, averaging non-uniform spectral line broadening with use of phase-alternated pulse sequence (PAPS), nutational spectroscopy.

The digital-to-analog conversion is implemented by an external 12-digit digital/analog converter AD9762. Filtration of side spectral components caused by nonlinearity of D/A converter operation is provided by a 7th order elliptic analog low-pass filter with the cutoff frequency ≈ 70 MHz. The amplitude of generated oscillations is controlled by an amplifier with LMH6504 voltage control, whose gain is assigned by a digital potentiometer operated with FPGA along a control bus. Matching of the device output resistance with the input resistance of a high-frequency power amplifier is done using an operational amplifier AD8009 with the band-pass of 1 GHz. Interface ports JTAG and Active Serial are provided for loading the program algorithm into the configuration memory. The device front panel has coaxial connectors for connecting transmitter and receiver paths of the radio-spectrometer, the

indicators of synthesizer operation and data transfer. To exchange data with the computer through USB interface, a hardware USB-UART controller FT232R is used.

The developed spectrometer system board (Fig. 8) provides not only pulse sequence shaping, but also implementation of the functions of NQR response processing and full control of the functional units of NQR Fourier spectrometer. In particular, a channel for analog-to-digital conversion based on an AD converter AD9280 and a matching amplifier AD8021 is provided for implementation of digital synchronous detection and subsequent digital processing of NQR response signals, as well as TTL-compatible channels using high-speed logic buffers 74LVC244.

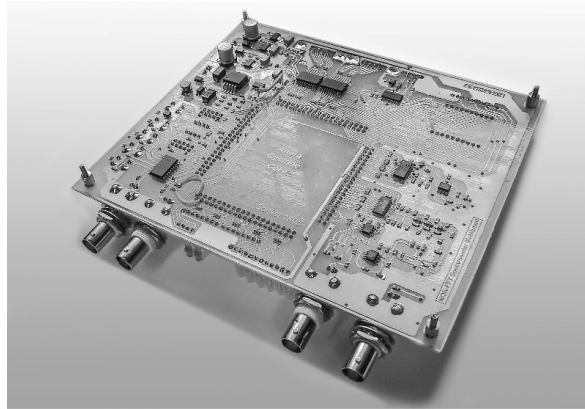


Fig. 8. The spectrometer system board.

The device was studied both in the single-pulse and multi-pulse modes. Fig. 9 shows the voltage oscillograms at the output of device when shaping some sequences most commonly encountered in NQR. The range of operating frequencies of the elaborated device was selected as 1–50 MHz, restricted to NQR frequencies of scientifically relevant isotope cores ^{14}N , ^{35}Cl , ^{63}Cu , ^{69}Ga , ^{71}Ga , ^{113}In , ^{115}In , etc.

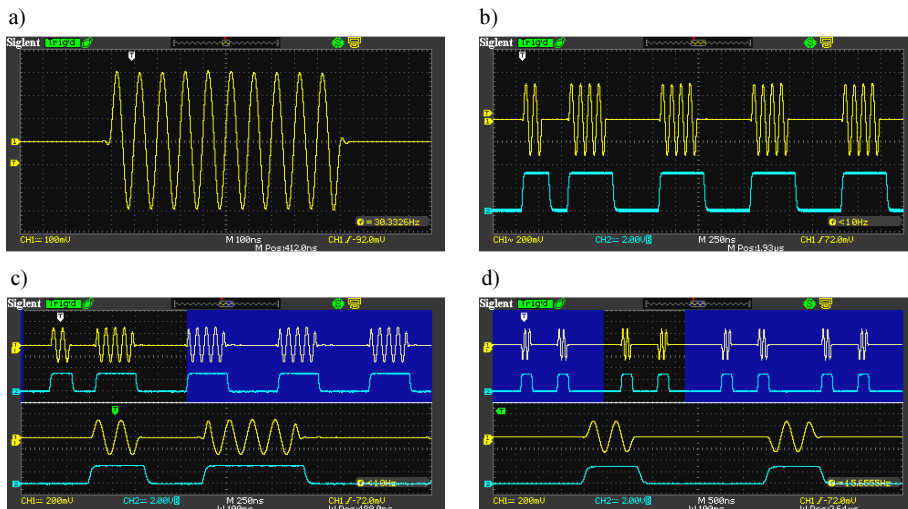


Fig. 9. The voltage oscillograms at the output of pulse sequence shaper: a) single RF pulse; b) Carl-Purcell; c) Meiboom-Gill; d) MREV-8 (WAHUHA).

5. Conclusions

A multi-functional programmer of pulse sequences for the NQR Fourier spectrometer has been developed on the basis of FPGA. The experimental test results have shown that the device assures shaping of pulsed sequences necessary for the pursuance of relaxation research in the frequency range of 1÷50 MHz.

The proposed programmer allows shaping of one-, two- and multi-pulsed sequences which enables the experiments on measuring relaxation times using the methods of Hahn, Carr-Purcell, Meiboom-Gill, *et al.* Owing to a wide range of changes in pulse lengths and the possibility of shaping various sequences, this programmer assures changes in parameters T_1 , T_2 , T_2^* .

Digital shaping of pulse sequences implemented in the proposed product assures high stability and accuracy of all temporal relations. The programmer guarantees phase binding of high-frequency filling of video pulses to pulse fronts.

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