Appl. Magn. Reson. 22, 455-473 (2002)

Applied Magnetic Resonance © Springer-Verlag 2002 Printed in Austria

# Application of Time-Variable Feedback to the Input Amplifier of Pulse Magnetic Resonance Spectrometers: Experimental Studies

A. V. Koptioug<sup>1</sup>, E. J. Reijerse<sup>2</sup>, and A. A. K. Klaassen<sup>2</sup>

<sup>1</sup>Institute of Chemical Kinetics and Combustion, Russian Academy of Sciences, Novosibirsk, Russian Federation <sup>2</sup>Department of Molecular Spectroscopy, University of Nijmegen, Nijmegen,

The Netherlands

Received August 29, 2001

Abstract. Experimental research on the improvement of the sensitivity and time resolution of pulsed magnetic resonance spectrometers is discussed. It is shown that application of a time-variable feedback of a signal to the input of the receiver amplifier can decouple the "fixed" relationship between the quality factor Q and the ringdown time of the resonance system. Experiments were performed with low-frequency, radio-frequency and microwave pulse-type magnetic resonance receivers. Modifications of an S/C-band electron spin echo modulation spectrometer carried out to check the "time-variable feedback" performance are described. It is demonstrated that the application of a time-variable feedback can significantly reduce the ringdown time and improve the recovery properties of the magnetic resonance receiver system. It is also demonstrated that the time-variable feedback can improve the overall receiver sensitivity due to the fact that the working bandwidth of the resonance system can be optimized separately for the transmitting and the receiving mode. Signal values could be increased at least three times and the signal-to-noise ratio about 1.5-2 times. The largest improvement is achieved with the initially overcoupled resonator. Experimental spectra of test samples for different settings of the time-variable feedback are shown.

## **1** Introduction

Improvement of the time resolution of pulse magnetic resonance instruments is an important design issue [1-3]. Decreasing the so-called dead time of the receiver has a profound influence upon the overall system performance, including its sensitivity [4-8]. Since induction signals decay exponentially, the ringing of the resonance system is "covering" the most intense initial part of the signal in question and therefore often spoils the precision of measurements. The problem seems to be general for all pulse-type receivers, including magnetic resonance techniques, and was widely studied for electron paramagnetic resonance (EPR) and nuclear magnetic resonance (NMR) applications in particular [9-38]. To obtain a high sensitivity of the magnetic resonance instrument, it is essential that the resonance element should have a relatively high quality factor Q. On the one hand, it governs the "efficiency" of transformation of microwave energy into the magnetic field causing the resonance transitions. Secondly, it governs the efficiency of "back-transformation" of the induced rotating spin magnetization into the radio-frequency (microwave) signal in the feeding line [7-11]. The quality factor thus strongly influences the signal intensity at the receiver input and the overall instrument sensitivity. On the other hand, for pulse-type instruments, where the measuring and pumping periods are separated in time, the resonator<sup>1</sup> quality factor should not be very high, as it prevents to achieve higher time resolution especially with the detection of free induction decay signals [1-6]. Moreover, with high-O resonance elements, short pumping pulse envelopes become distorted (due to bandwidth limiting by the resonator) and the resonance system tends to "ring" after each pulse. Excess ringing after the pumping pulse forces the receiver to be switched on with a delay, which leads to a loss of the free induction decay signal intensity [3, 10, 11, 15–17, 22, 23, 28, 38]. Even with the detection of echo signals, which themselves are delayed after the pumping pulse, excess ringing can still cause problems and lead to the decay of instrument sensitivity. Finally, the reduced bandwidth of the resonance structure not only causes distortions of the pumping pulse but also often prevents uniform excitation of the magnetic resonance spectrum under study. From these points of view, relatively low-Q resonators could seem to be more favorable (this subject is discussed in detail in ref. 38). The trends discussed above usually do not allow combining top sensitivity with the best time resolution in pulse spectrometers employing traditional reflection resonance architectures. Commonly a certain compromise between the highest possible sensitivity and the shortest possible ringing time of the resonance system is chosen depending on the particular research tasks.

One of the best ways out of the above "high versus low Q dilemma" is the detection of echo signals instead of free induction decay [3]. But when the detection of free induction decay signal is essential, some other solutions are to be applied. Among these are shunting of the resonance circuit (either directly, by a parallel active switch [18–23], or by short-circuiting a coil magnetically coupled to the main resonance system [29, 30, 33]) after the pumping pulse, which is often used with the radio-frequency systems. Unfortunately the dumping by active switching is not easy to implement for microwave spectrometers and systems with high pumping pulse power. Another option is the implementation of so-called "crossed-field" type probes [32–37] constructed of two resonances with orthogonal magnetic fields. These systems are extremely complicated, as they depend on the precise simultaneous tuning of two resonances (both resonance frequency and coupling) with precisely orthogonal magnetic fields. Even the introduction of a sample tends to change the balance and leads to lengthy read-

<sup>&</sup>lt;sup>1</sup> The terms "resonator", "resonance system" and "resonance circuit" are used here in a wide sense, covering lumped LC circuits as well as quasi-lumped structures and volume cavities.

justments each time it is done. Still these probes allow much better signal recovery due to the fact that "channel-to-channel" coupling in the well-tuned crossfield system exists mainly through the sample magnetization. The implementation of cross-field probes could be regarded as a way of spatial separation of the excitation and induced radio-frequency magnetic and electric fields, minimizing the signal transfer from the transmitting resonance element (Tx channel) into the receiving one (Rx channel). In some sense the use of cross-field type probes in pulsed spectrometers complements the time domain separation of pumping and received signals by the "spatial" separation (orthogonality) of the radio-frequency (microwave) fields of the Tx and Rx channels. Implementation of cross-field probes significantly reduces direct penetration of the ringing signal from the excitation resonator into the receiver. It also substantially decreases the noise contribution of the oscillator frequency and phase noise as compared to the simple reflection resonator diagrams [30–37, 39, 40]. Thus its only significant drawback seems to be technical complexity.

With pulse EPR (spin-echo) spectrometers all discussed technical problems are enhanced due to the specifics of microwave instrumentation design. Resonator dumping by parallel active shunts during or after the excitation pulses is extremely problematic as pumping pulses often have a peak power in the kilowatt range and volume cavity resonators may have quality factors up to tens of thousands. So the demands on active shunt elements (breakdown voltage, losses in open and closed states, working frequency range, etc.) are extremely tight. Cross-field-type probes in the microwave region are quite complex and not very effective [34–36] mainly due to the significance of the always present parasitic capacitive coupling between the Rx and Tx channel elements of the probe system. Quite often the perfect tuning of the cross-field probe is completely destroyed by the sample placed into it. Thus, to tackle the microwave resonator ringing, some other solutions should be found.

First let us discuss the major sources of resonator ringing. Extensive research has shown two basic sources of "parasitic ringing" in reflection-resonator-type instruments. The first source is the resonator itself, as the signal is developing and falling exponentially with the characteristic time equal to  $Q_L$  periods of resonance frequency [8, 9, 41]:

$$\tau = Q_{\rm L}/F_0,$$

where  $Q_L$  is a "loaded quality factor" (quality factor of the resonance system with the feeding line and everything else coupled to it). A high- $Q_L$  resonator always has a long ringdown time after the pumping pulse, allowing all the energy stored during the pumping pulse to dissipate in the resonator itself and the structures coupled to it. In some cases nonresonant ( $Q_L \approx 1$ ) probes, mainly transmission-line-based, could be used providing very high time resolution performance [18, 19, 42]. But the poor sensitivity associated with these extremely low-Qprobes strongly reduces their field of application.

Other sources of ringing were found to be of acoustic and eddy current origin [43-48]. Acoustic type ringing is associated with standing acoustic waves in

the mechanical construction. This ringing is caused by the magneto- or electrostriction excited in the resonance system during the high-power pulses. Vibrations lead to the mechanical distortion of the resonator shape, and coupling structure shape and position. Consequently it leads to an offset in the resonance frequency and coupling depth and thus to the arrival of additional noise at the receiver. The latter effect is most pronounced for bridge-type continuous-wave (cw) spectrometer systems, but could also contribute to the resonator ringing in a pulse system as well. It is known that mechanical resonance systems can have extremely high quality factors, so the corresponding acoustic induced ringing could potentially cause problems for up to a few seconds after the pumping pulse. Another type of eddy-current-related ringing with a complex nature is attributed both to the acoustic and electromagnetic effects. During the excitation pulse, delivering substantial energy to the resonance system in a short period of time, strong currents are induced all over the probe, including the resonator "body" as well as the screen ("mirror currents"). Eddy currents, induced in the screen, could have a substantial decay time and are picked up by the resonance element producing substantial parasitic signals along with the acoustic vibrations.

As the sources of the parasitic ringing mentioned above are essentially different, the ways of their suppression also differ. Acoustic and eddy-current-induced ringing suppression is achieved via the careful mechanical design of the resonance system including the proper construction-material selection, special design of the holders, wire-wound or slotted screens, etc., turning it into the domain of the fine art rather than science [45–49]. The ringing caused by the dissipation of the energy in resonator itself cannot be easily eliminated. One of the possibilities to increase the time resolution of the system is mechanical variation of the resonator coupling.

To provide the highest power transfer ratio from the transmitter to the resonance element via the transmission feeder line and coupling structure, the matching (critical coupling) condition is to be met [38, 50–55]. It occurs when the resonance impedance of the cavity transformed by the coupling system becomes equal to the characteristic impedance of the transmission line (providing that it is in turn equal to the input impedance of the receiver<sup>2</sup>). Under these conditions the loaded quality factor of the resonance element with critical coupling is half that of the unloaded one [38, 41]:

$$Q_{\rm L}=1/2Q_0.$$

It corresponds to the case when the energy is equally divided between the resonator and transmitter output during the pumping cycle (or resonator and the receiver input during reception). Increasing the coupling depth over the critical value in the reception mode is equivalent to a stronger coupling of the resonator to

<sup>&</sup>lt;sup>2</sup> The term "receiver" is used here for a whole assembly comprising the input resonance circuit, coupling structure, input amplifier and the feedback (if present).

external resistors, forcing more energy to leak out and dissipate there faster. Usually it also means a decrease in the signal amplitude at the receiver (for a fixed delay time and the same pumping power), since the mechanical coupling element<sup>3</sup> cannot be readjusted fast enough to provide optimum conditions for the reception if the optimum coupling for the transmission mode is above critical. At the same time, however, the reduced ringing time of the overcoupled structure leads to the possibility of signal detection at shorter delay times. It was experimentally shown that a good compromise in a trade of sensitivity versus dead time with reflection resonance system spectrometers is reached by choosing the resonance element coupling somewhat larger than the critical value [16, 38, 53–55].

The most promising alternative option, which can be equally helpful for both radio-frequency and microwave pulsed spectrometers, is the application of a "quenching pulse" after the last pumping pulse in the sequence [12–15, 23]. If a signal shifted in phase exactly 180° is applied immediately after the pumping pulse, resonator ringing can be effectively suppressed (quenched). In the micro-wave systems this is achieved either with special "by-pass" delay lines [12] or including a special quenching-pulse-forming branch [13, 14] into the system, while in radio-frequency systems it is usually achieved via so-called bi-phase modulators [15, 23]. The main problem with this technique is the difficult choice of amplitude and duration of the quenching pulse: improperly adjusted it could cause additional ringing, rather than quenching of the existing unwanted signal [23].

The overall pulse spectrometer sensitivity dependence on the resonance system coupling depth and quality factor of the resonator is not a priori clear. Extensive research was carried out for various spectrometer configurations [8-11, 17, 23, 39, 40, 52, 56-62] to find out the best conditions. It was found that an overcoupled resonance system (coupling stronger than critical) might be not exactly optimal for the signal reception mode if ringing problems are disregarded ("reception mode only" case). This effect is mainly attributed to the fact that amplifier noise performance is very sensitive to the output impedance of the signal source. It was also shown that an overcoupled resonator with high unloaded Qprovides a better signal-to-noise (S/N) ratio in the pulse magnetic resonance experiment, as compared to the one with the same volume, working frequency and critically coupled to the same loaded quality factor but with lower unloaded Q[38]. However, the dead time problems still occur even in the case of mechanically overcoupled resonators. Thus the discrepancy between a short dead time and the highest possible S/N ratio still stays when choosing either under-, overor critically coupled resonance system in the reflection type spectrometer.

If mechanically fixed overcoupling still does not solve the problem, the time variation of some other parameters might possibly do it. The introduction of

<sup>&</sup>lt;sup>3</sup> The term "mechanical coupling element" is used here to designate a part of the construction such as wave guide iris, coupling loop, capacitive rod; its variation invokes some mechanical movement – translation or rotation of adjustment screws, etc.

electronically controlled feedback into the receiver could in fact provide an alternative solution [63, 64]. It is known that negative feedback decreases the effective input impedance of the amplifier [65]. In turn, the amplifier input shunts the resonator causing a decrease in the effective quality factor, which is exactly what is necessary for the suppression of resonator ringing. The application of the quenching pulse [12–15, 23] could be regarded as an application of the negative feedback for a short time after the pumping pulse, and this feedback is implemented through the pumping channel of spectrometer. But the time-variable feedback could be implemented in the receiver channel as well. Negative feedback could be invoked to suppress the residual resonator ringing. After the effective ringing suppression a positive feedback could be activated to restore the lower receiver bandwidth and provide higher sensitivity. This can be achieved because the time-variable feedback allows changes in the effective receiver bandwidth without mechanically readjusting resonator coupling mechanisms.

It should be mentioned here that a variable-feedback approach was in fact extensively used in the early days of the radio reception and is still in use for the superregenerative type receivers [66-69] and front-end amplifiers with "quality factor magnification" [70-77]. But the development of low-noise semiconductor front-end devices presently led to the deflection of the designers' interest towards the alternative receiver architectures. In certain cases, magnetic resonance systems with superregenerative receivers are still in use, in particular for the nuclear quadrupole resonance (NQR) relaxation measurements [65, 67, 73-75]. In recent years the interest in the idea of time-variable positive-negative feedback combination has returned, in particular for the application to the miniature narrow-band amplifiers and digital filters [76-80]. Negative cw feedback is actually used in the input amplifiers to regulate the effective receiver bandwidth [30, 31]. Input amplifiers with a cw (not variable in time) feedback are also used in high-resolution NMR to eliminate the effect of the signal frequency shift caused by the resonator-to-spin system coupling [43-45]. This effect is due to the interaction of the spin system "resonator" with the magnetic resonance probe and is analogous to the frequency shift of the individual resonance in coupled LC circuits caused by mutual coupling. In practice, in any amplifier a whole set of positive-negative feedback loops is present either intentionally or parasitic. But the time-variable feedback application in the receivers is suggested here for the first time.

#### 2 Results and Discussion

For the purpose of the current research it is worth returning to the discussion on the quality factor "magnification" principles and the feedback manipulation. In the case of an amplifier with the feedback connected to the resonance circuit (Fig. 1) the overall signal transfer coefficient strongly depends not only upon the resonator unloaded quality factor and the coupling ratio but also on the feedback parameters and the amplifier input impedance value. It has been shown that the losses in the sample could be taken into account by increasing the resistor

460



Fig. 1. Principal block diagram of the magnetic resonance receiver comprising the resonance element with coupling structure and input amplifier with two-branch feedback system controlled by switches.

value representing the losses in the resonance circuit equivalent diagram [81–84] and thereby effectively decreasing the resonator quality factor. So one has in fact all components of the simplified equivalent diagram modelling the receiver system.

If the input impedance of the amplifier is taken to be purely active and the feedback is purely resistive and not extremely strong (does not cause a significant shift of the overall resonance frequency), then the action of the feedback could be summarized as follows. Firstly, the resonance frequency of the critically loaded LC circuit is lower than that of the stand-alone circuit  $F_0$  (case of extremely weak coupling). Application of positive feedback in the amplifier shifts the resonance frequency up (towards  $F_0$ ) and decreases the "effective working bandwidth", which is interpreted as an increase in the effective quality factor of the system. Negative feedback shifts the resonance frequency down (further from  $F_0$ ) and increases the effective working bandwidth [30, 31, 85], which is interpreted as a decrease in the effective quality factor. Strong positive feedback could make the effective quality factor even significantly higher than the unloaded quality factor of the stand-alone resonator (effect of Q factor magnification [70– 72, 75, 78]). It is generally assumed that with the traditional cw resonance receivers, variation of the bandwidth by changing feedback does not improve the resulting S/N ratio at the output [30, 31, 75, 85]. So only the variation of the feedback in time could possibly bring some improvements.

In reality, however, amplifier and feedback parameters are often essentially frequency-dependent, and the employed simple reasoning might fail. Experiments were performed to support the above general claims and to show that qualitative conclusions drawn from a simplistic model could be used for receiver systems operating at very different frequencies. For the first tests a cw longitudinally detected ESR (LODESR) [86–89] low-frequency receiver working around a few

hundreds of kilohertz was used. In the case of cw low-field LODESR the pumping of EPR transitions is carried out at radio frequencies (hundreds of megahertz) and the signal is picked up at a comparatively low modulation frequency (from tens to hundreds of kilohertz) [88, 89]. Significant frequency offset between the Tx and Rx channels allows necessary manipulations with the receiver to be easily carried out. The principal diagram for the tests carried out with the LODESR receiver is presented in Fig. 2. Initially (the coupling branch  $R_F$ ,  $C_F$  is disconnected) the capacitive divider  $C_1$ ,  $C_2$  was adjusted to provide a critical coupling to the resonance circuit. To check the receiver performance, the network analyzer output was weakly inductively coupled to the coil and its input connected to the amplifier output. The existing input amplifier was only slightly modified to provide the possibility of additional signal inversion (by flipping a single switch) without changing the actual amplification rate at the working frequency. The activation of the feedback was causing the expected changes in the amplification rate versus frequency and phase versus frequency dependences for the receiver system. It was possible to change the effective quality factor (calculated as the ratio of resonance frequency to the effective bandwidth, measured at the -3 dB level) between 2 and 900. The initial effective Q value was about 120 for the LC circuit with a resonance frequency of 250 kHz optimally coupled to the high input impedance (JFET) amplifier with no feedback. The resonance frequency (maximum of the amplitude versus frequency or zero passing of the phase versus frequency dependence) shift in the case of positive feedback appears to be more complex than expected from the simplistic model. Most probably this



Fig. 2. Principal diagram of the low-frequency cw measurements of the LODESR receiver effective bandwidth. Input amplifier primary stage is performed with the JFET J309 from Siliconix Inc., Santa Clara, CA, USA;  $K_1 = 12$ . Basic amplifier is constructed with low-noise operational amplifiers AD797 and OP80 from Analog Devices Inc., San Jose, CA, USA;  $K_2 = \pm 200$ . Overall input amplifier bandwidth, 500 kHz. Network analyzer, MS3606B from Anritsu Ltd., Luton, UK. Measurements were performed for the resonance frequencies of 100 to 450 kHz.

is due to the fact that feedback application causes changes in the effective coupling depth and the amplifier phase shift is frequency-dependent. Still the resonance position clearly shifts towards the value for the stand-alone resonance circuit. Of course, a change in the phase shift across the feedback or additional phase shift in the amplifier could lead to the additional shift of the new resonance position within the initial resonance circuit "bandwidth" up or down the frequency. It is due to the fact that the phase-balance condition in the discussed receiver is at least as important as the amplitude-balance condition. Also a strong positive feedback could not provide very large enhancement of the effective quality factor as the stability of the described receiver system is compromised and it starts to self-oscillate.

As it was expected, cw LODESR experiments performed with the discussed variable-feedback receiver have shown no improvement in the S/N ratio. This is attributed primarily to the fact that with the used LODESR setup the main amplification is carried out in the lock-in receiver placed after the input variable-feedback unit. In this case the lock-in receiver determines the overall receiver bandwidth (about 1 to 10 Hz as defined by the time constant) [78, 87–89]. The resonance LC circuit effective bandwidth even with the highest possible Q magnification was not smaller than about 50 Hz, so it was always much larger than that of the lock-in receiver.

Thus, the main conclusion is that the variable-feedback input amplifier is not effective in the case of cw spectrometers, though it does provide a good means to vary the receiver bandwidth without much degrading the S/N ratio. But in the case of pulse spectrometers the situation is different. It should be noted that switching to a new quality factor value when switching feedback in the input amplifier could be regarded as instantaneous (or occurring with the time of switch on-off toggling). It could seem strange, but one should not forget that changes in signal amplitude at the output of a resonance amplifier are exponential, with the time constants determined by the final effective quality factor value, which is not instantaneous at all. So, to detect a difference at the receiver output, it should take about  $Q_{\rm eff}$  periods for the signal intensity to change e times.

To illustrate the time switching of the effective quality factor in the receiver with a time-variable feedback, experiments were also performed with receivers operating at different frequencies in the 1–100 MHz range. The basic principal diagram of the setup is presented in Fig. 3. The resonance LC circuit (1) is weakly coupled to the signal generator (8) and critically coupled to the low-noise amplifier (2) via a 50  $\Omega$  cable line (6). Both coupling elements of the resonance circuit are depicted as inductive, though other types of coupling were tried out as well. Different types of low-noise amplifier input stages were tried, including bipolar transistors, JFETs and operational amplifiers. For the lower frequencies an amplifier with a JFET input and two consecutive balance cascade sections (with matched transistor pairs MAT-01 from Analog Devices Inc.) was used. The overall bandwidth of this amplifier was 0.1–25 MHz, and the overall gain amounted to about 900. For the higher frequencies, various radio-frequency amplifier chips were tried. The feedback section contains two identical variable-gain amplifier sec-



Fig. 3. Principal diagram of the radio-frequency pulse mode measurements of the effective quality factor for resonance receivers. (1) Resonance element with coupling structures; (2) input low-noise amplifier; (3) output buffer; (4) two variable-gain amplifier stages controlling the positive and negative feedback timing and depth; (5) matching transformer; (6) coaxial transmission line connecting the probe (10) and amplifier assembly (9); (7) coupling structures; (8) signal generator weakly coupled to the resonance circuit.

tions (4) and a matching transformer (5). It was found that satisfactory switching time (about 0.5  $\mu$ s or less) and reasonably low level of glitches could be achieved with the variable gain amplifier chips VCA-610 (by Burr-Brown Inc., Tucson, AZ). The feedback level is easily controlled then by varying the amplitude of the pulses supplied to the gain control pin of the VCA-610 chips.

The timing diagram of a typical variable-feedback experiment performed in the frequency range of 1.2-13 MHz is illustrated in Fig. 4. Positive feedback with variable depth was switched on for an interval of 10 to 100  $\mu$ s (Q magnification mode). Negative feedback was turned on for a shorter period, when no positive feedback was invoked (O suppression mode). The repetition rate of the whole sequence was 1 to 5 kHz. The timing sequence was designed to allow a period without feedback to be followed by a period when positive feedback was active, then a period without feedback and finally a period when negative feedback was active. Offsetting the generator frequency from the initial value it is easy to measure the effective resonance curves for the overall receiver system for all three states of the system by measuring the amplitude of the signal during the periods of positive feedback, negative feedback and no feedback. For the employed moderate-O resonance circuits the corresponding effective loaded quality factors without the feedback (measured as the ratio of the width of the overall receiver resonance curve at half-power height to the central frequency value) were about 40-80. When negative feedback was activated, the effective Q value was reduced to 1.5-5, depending on the feedback level. When positive feedback was

Time-Variable Feedback in Magnetic Resonance: Experimental



Fig. 4. Feedback timing and the signal envelope development for the time-variable feedback experiments. Top: signal envelope at the output of the receiver. Middle: timing diagrams for the positive-feedback-variable gain amplifier and for the negative-feedback-variable gain amplifier. Bottom: the receiver amplification rate versus frequency dependence as measured at different time intervals during the feedback timing sequence within the  $Q_1$ , positive feedback invoked;  $Q_2$ , no feedback;  $Q_3$ , negative feedback invoked.

activated, the effective Q value was boosted to about 200–250. A larger increase in the effective quality factor value was possible but problematic mainly due to receiver instabilities. The changes in the effective quality factor value were accompanied by the corresponding changes in the signal amplitude rise and decay times (top trace in Fig. 4) and phase shifts. Note here that when the negative feedback is activated, the signal magnitude decay is very fast (characteristic time constant  $\tau_3$  in Fig. 4) as it is governed by the extremely low effective quality factor  $(Q_3)$ . When the positive feedback is activated, the signal magnitude is increasing with a much larger time constant  $(\tau_i)$  corresponding to the magnified quality factor value  $(Q_1)$ . The transient behavior of the signal when turning to the state without the feedback is governed by the initial effective quality factor value ( $Q_2$ ,  $\tau_2$  in Fig. 4). Such a complex timing diagram was chosen deliberately to demonstrate the action of the time-variable (level and type) feedback upon the receiver performance. For the common pulse receiver systems it is feasible to use a simpler timing scheme when either negative feedback in the input amplifier is active (suppression of the residual resonance system ringing) or when positive feedback is active (signal acquisition, quality-factor magnification).

Unlike the cw technique, in the pulse magnetic resonance spectrometers the input amplifier feedback manipulation could provide the improvement in the overall S/N ratio and the reduction of the dead time as well. This could be achieved by adjusting the optimum coupling conditions for Tx and Rx modes independently. To prove this, experiments were conducted on a modified S/Cband electron spin-echo spectrometer [90]. In the case of spin-echo spectrometers, amplifiers in the receiver channel are usually wideband, as they need to reproduce correctly the free induction decay and echo signal envelopes. In addition, the used microwave amplifiers commonly have larger noise values compared to the radio-frequency ones. In this case, the application of positive feedback in the input amplifier leading to the smaller effective bandwidth during the reception could be beneficial. The diagram of the modified reception channel of the spin echo spectrometer is presented in Fig. 5. Basic tests were performed with the small-volume microwave Alderman Grant resonators [91, 92] having loaded quality factors in the range of 350-500 (critically coupled). No additional elements were inserted into the main receiver branch except a coupler (8) after the low-noise amplifier. Also the two outputs of the directional coupler used for reflected signal monitoring (2) were connected in an unusual way. The feedback arm was constructed analogously to the one of the low-power part in the pumping-pulse former branch. It consists of the isolator (11), pin-diode switch (12), attenuator (13) and a phase shifter (14). This configuration allows either positive



Fig. 5. Principal diagram of the modified S/C-band spin echo spectrometer used for the time-variable feedback experiments: (1)-(10) receiver (signal) arm, (11)-(14) additional (feedback) arm. (1) Resonator; (2) directional coupler (-6 dB); (3) circulator; (4, 5) receiver protection pin-switches; (6) low-noise input amplifier (+20 dB); (7) isolator; (8) directional coupler (-6 dB); (9) mixer; (10) PCC liber (12) to the (12) Couple (-6) dB); (7) isolator; (8) directional coupler (-6 dB); (9) mixer; (10)

DC block; (11) isolator; (12) feedback timing pin-switch; (13) attenuator; (14) phase shifter.

466

or negative feedback applied to the resonator depending on the phase settings. The feedback branch could be also completely blocked by the pin-switch (12).

The basic timing diagram of the "variable-feedback" experiment is presented in Fig. 6a. Spin-echo signals were detected with the usual two-pulse sequences. A feedback-blocking switch was opened either simultaneously with the gating of the receiver channel by the protection switches or after a small delay. Field swept spin-echo detected EPR spectra and echo envelopes were detected with the boxcar averaging system [90]. Characteristic shapes of the signal envelopes after the video amplifier are schematically depicted in Fig. 6b for the various feedback phase shifter and attenuator settings. Negative feedback resulted in a reduction of the echo signals, noise as well as residual ringing (trace 8), as expected. Positive feedback was capable of increasing the echo envelope in some cases up to 6-10 dB (trace 9). A further increase in the positive feedback depth was leading first to a stochastic instability of the receiver system (trace 10) and then to the continuous self-oscillation of the receiver during the whole period of the feedback operation. The same setting of the feedback branch attenuator enabled very deep suppression of the echo envelope and residual ringing when changing the feedback branch phase shifter setting, turning from almost 100% positive to almost 100% negative feedback. Figures 7 and 8 present the sets of ESE-detected EPR spectra of the test sample (coal) with feedback channel switched off (middle



Fig. 6. a Pulse timing diagram for the time-variable feedback-type spin-echo experiments. Common two-pulse sequence without phase altering and quenching pulses was used. (1) Pumping pulses envelope after the detector; (2) receiver protection switch timing  $(D_1, \text{ delay of the receiver "on" switching after the last pumping pulse); (3) feedback switch timing <math>(D_2, \text{ delay of the feedback activation after the receiver "on" switching); (4) video signal envelope after the mixer; (5) residual resonator ringing signal; (6) spin echo signal. b Characteristic shape of the output signals for: (7) overcoupled resonator, feedback is off; (8) strong negative feedback is on, both the cavity ringing and the main signal are suppressed; (9) positive feedback; attenuator setting close to the critical value: signal envelope is distorted due to the instabilities and sporadic self-oscillation development.$ 

trace), with positive (upper trace) and negative (lower trace) feedback on, when the experiment timing is set as described above. Signal values in some cases could be increased at least three times, while the S/N ratio was growing 1.5-2 times at best. The largest difference in the echo signal with feedback alternation was achieved with the initially overcoupled resonator (here initially means the coupling of the resonator set without activation of the feedback). The smallest difference occurred for the resonator initially coupled critically or undercoupled. when almost no S/N ratio improvement was achieved by applying the positive feedback. Such behavior most probably indicates that the input amplifier noise contribution rather than the resonator one dominates the noise in the system (though extensive theoretical studies are needed to explain this effect in detail). Thus the application of the positive feedback can improve the S/N ratio, when effectively switching from the overcoupled to the critically coupled resonator. Application of the feedback with the input amplifier critically coupled to the resonator for a chosen receiver configuration only adds more noise. Therefore the use of positive feedback for reflection resonator type ESE spectrometers with dielectric resonators, which usually have extremely high unloaded quality factors [93-95] and thus are generally strongly overcoupled, could bring significant improvement in their S/N performance.

It looks hardly realistic that the quality factor magnification by positive feedback could increase the echo signal amplitudes by more than 10–15 dB even with



Fig. 7. Spin-echo detected EPR spectra of the coal sample (from Bruker BioSpin GmbH, Karlsruhe, Germany) taken at room temperature. Microwave Alderman Grant resonator [91, 92]; resonance frequency, 4.77 GHz; unloaded quality factor  $Q_0 = 400$ . Resonator was overcoupled to provide short ringing time. Field sweep rate, 250 G per minute; central field, 1700 G. Boxcar amplifier Princeton 162 from EG&G Instruments, Princeton, NJ, USA; averaging window, 10  $\mu$ s. Two-pulse pumping sequence; duration of the pulses, 30 and 60 ns; acquisition delay time,  $\tau = 520$  ns; repetition rate, 10 kHz. Upper trace, positive feedback is on; middle trace, feedback is off; lower trace, negative feedback is on. Insets show the noise part of the trace multiplied 4 times.

468



Fig. 8. Spin-echo-detected ESR spectra of the coal sample (from Bruker BioSpin, Karlsruhe, Germany) taken at room temperature. Overcoupled microwave Alderman-Grant resonator; resonance frequency, 4.74 GHz; field sweep rate, 250 G per minute; boxcar amplifier averaging window, 10  $\mu$ s. Two-pulse pumping sequence; repetition rate, 10 kHz. Left traces: duration of the pulses, 30 and 60 ns; acquisition delay time,  $\tau = 550$  ns. Right traces: duration of the pulses, 30 and 60 ns; acquisition delay time,  $\tau = 450$  ns. Upper traces, positive feedback is on; middle traces, feedback is off; lower traces, negative feedback is on.

an extremely stable amplification system. The main reason seems to be the instabilities in the input amplifier phase parameters. Also it may appear unreasonable to do so because when the corresponding bandwidth becomes too small it causes distortions in the spin echo signal envelope. In the case of very strong feedback the resonance frequency shift of the overall receiver system becomes significant as well, causing additional problems with system tuning. But the influence of the discussed factors upon instrument performance and ESE spectra (usually having rather wide lines) has yet to be evaluated. For example, the frequency shift caused by switching from strong resonator overcoupling to critical coupling could be even successfully utilized for complex systems with offset pumping and reception frequencies.

To achieve both positive and negative time-variable feedback applied in the same experiment, two separate feedback branches are necessary. Despite the higher complexity of such a system, it could be recommended for advanced pulsed magnetic resonance spectrometers. The most benefits of such a system could be expected when time-variable feedback will be used along with the quenching pulse implementation. In this case the input amplifier could be disconnected from the resonator during the pumping pulse, but negative feedback could stay on during this time (see Fig. 9). Negative feedback should be disabled somewhat later than the amplifier protection switches connecting the amplifier to the system. This should allow for effective suppression of the switching transients along with the residual resonator ringing. Positive feedback is dis-

abled to allow effective signal acquisition. As positive feedback increases the effective amplifier input impedance, it could change the effective coupling to a resonance circuit to the desired critical or even undercoupled levels. Thus, time variable feedback could allow changing the effective coupling without mechanical changes in the resonator coupling structure. As effective coupling control here is achieved electronically in the low-power channel, this could provide conditions close to optimal for both the pumping (Tx) and reception (Rx) modes for the resonant system with mechanically fixed coupling and tuning.

It is also clear that best results with the time-variable feedback implementation in the pulsed magnetic resonance spectrometers could be achieved for high Q, strongly overcoupled resonators. In such situation one should apply the term "restoration" rather than magnification of the quality factor. When overcoupling an initially high-Q resonator, the application of the feedback to restore the Q value should cause no problems with the stability of the system. Also the resonance amplifier with negative feedback is generally more stable to input overloads and pro-



Fig. 9. Suggested pulse sequence timing diagram for the time-variable feedback-type magnetic resonance experiments. Common two-pulse spin-echo sequence with the quenching pulse is shown as an example. (1) Pumping pulse envelope after the detector (negative signal indicates the 180° phase shift of the quenching pulse); (2) quenching pulse timing; (3) receiver protection switch timing; (4) negative feedback switch timing; (5) positive feedback switch timing; (6) signal envelope after the mixer; (7, 8) residual pick-up signal from the pumping pulses; (9) residual pick-up signal from the quenching pulse; (10) residual resonator ringing signal and switching pulse glitches; (11) spin-echo signal.

vides shorter recovery times. The frequency shift of the effective resonance with correct resonator and amplifier parameter choice becomes less important, as the effective bandwidth becomes large and the new resonance curve covers the initial resonance frequency anyway (Fig. 4). It is very difficult to predict how the discussed system will work when jumping from strong resonator overcoupling to undercoupling conditions. Taking into account long sections of transmission line (coaxial or wave guide) connecting the resonance element to receiver assembly which at least produce some delays (phase shift), it could happen that the most effective would be to switch from the resonance system overcoupling to the critical coupling. Extensive research to answer these questions is under way.

### **3** Conclusions

No initial restrictions on working frequency were set when studying basic features of the time-variable feedback application in the input receivers. Thus qualitative conclusions should be valid for both radio-frequency and microwave receiver systems. Experiments with the receivers for magnetic resonance spectrometers confirmed that time-variable feedback could be successfully utilised to improve the system performance for pulsed resonance receivers with working frequencies from audio to microwave frequencies. In all cases, increasing the time resolution and improving the sensitivity of the pulsed resonance receivers and allowing the easy implementation of the variable bandwidth function could achieve the benefits. Separate optimization of the bandwidth and optimum resonator coupling for Tx and Rx modes, which could be achieved electronically by the input amplifier feedback control, is a definite advantage of the discussed technique. In general terms, implementation of the time-variable feedback in the resonance input amplifier could allow an easier way of system performance optimization.

#### Acknowledgement

A.V.K. is grateful to the Dutch Organization for Scientific Research (NWO) for supporting the research (grant 04713089).

#### References

- 1. Kevan L., Schwartz R.N.: Time Domain Electron Spin Resonance. New York: Wiley 1979.
- 2. Carrington A., McLachlan A.D.: Introduction to Magnetic Resonance with Applications to Chemistry and Chemical Physics. New York: Harper and Row 1967.
- 3. Cho H., Pfenniger S., Gemperle C., Schweiger A., Ernst R.R.: Chem. Phys. Lett. 60, 391-395 (1989)
- 4. Abragam A.: The Principles of Nuclear Magnetism. Oxford: Clarendon Press 1967.
- 5. Losche L.: Nuclear Induction. Berlin: Deutscher Verlag der Wissenschaften 1957.
- 6. Poole C.P. Jr.: Electron Spin Resonance: A Comprehensive Treatise and Experimental Technique, 2nd edn. New York: Wiley 1983.

- 7. Feher G.: Bell Syst. Technol. J. 36, 449-484 (1957)
- 8. Cho S.-I., Sullivan N.S.: Concepts Magn. Reson. 4, 227-243 (1992)
- 9. Cho S.-I., Sullivan N.S.: Concepts Magn. Reson. 4, 293-306 (1992)
- 10. Rinard G.A., Quine R.W., Song R., Eaton G.R., Eaton S.S.: J. Magn. Reson. 140, 69-83 (1999)
- 11. Rinard G.A., Quine R.W., Harbridge J.R., Song R., Eaton G.R., Eaton S.S.: J. Magn. Reson. 140, 218-227 (1999)
- 12. Davies J.L., Mims W.B.: Rev. Sci. Instrum. 52, 131-132 (1981)
- 13. Narayana P.A., Massoth R.J., Kevan L.: Rev. Sci. Instrum. 53, 624-626 (1982)
- 14. Holczer K.: Bruker Rep. 2, 40-41 (1986)
- 15. Rudakov T.N., Belyakov A.V., Mikhaltsevich V.T.: Meas. Sci. Technol. 8, 444-448 (1997)
- 16. Chingas G.C.: J. Magn. Reson. 54, 153-157 (1983)
- 17. Hoult D.I.: Prog. Nucl. Magn. Reson. Spectrosc. 12, 41-77 (1978)
- Khmelinskij V.E.: Russian Patent application N1174879 of 22.07.1967, Patent granting decision of 15.11.1968
- Lowe I.J., Engelsberg M.: Rev. Sci. Instrum. 45, 631–639 (1974); Lowe I.J., Whitson D.W.: Rev. Sci. Instrum. 48, 268–273 (1977)
- 20. Conradi M.S.: Rev. Sci. Instrum. 48, 359-361 (1977)
- 21. Kisman K.E., Armstrong R.L.: Rev. Sci. Instrum. 45, 1159-1163 (1974)
- 22. Garroway A.N., Ware D.: Rev. Sci. Instrum. 46, 1342-1343 (1975)
- 23. Hoult D.I.: Rev. Sci. Instrum. 50, 193-200 (1979)
- 24. Froncisz W., Jesmanowicz A., Hyde J.S.: J. Magn. Reson. 66, 135-143 (1986)
- 25. Reykowski A., Wright S.M., Porter J.R.: Magn. Reson. Med. 33, 848-852 (1995)
- 26. Pollak V.L., Slater R.R.: Rev. Sci. Instrum. 37, 268-272 (1966)
- 27. Kuhns P.L.: J. Magn. Reson. 78, 69-76 (1988)
- 28. Raad A., Darrasse L.: Magn. Reson. Imaging 10, 55-65 (1991)
- 29. Jurga K.: J. Phys. E Sci. Instrum. 14, 555-560 (1981)
- Luyten M.J., Korbee D.D., Claassen-Vujèic T., Melkopf A.F. in: Proceedings of the Society of Magnetic Resonance 3rd Meeting and European Society for Magnetic Resonance in Medicine and Biology 12th Meeting, p. 934, Nice, France, August 19-25, 1995.
- Borsboom H.M., Trommel J., Melkopf A.F. in: Proceedings of the Society of Magnetic Resonance 12th Annual Meeting, p. 1355, New York, USA August 14-20, 1993.
- Alecci M., Brivati J.A., Placidi G., Testa L., Lurie D.J., Sotgiu A.: J. Magn. Reson. 132, 162– 166 (1998)
- 33. Floridi G., Lamanna R., Cannistraro S.: Meas. Sci. Technol. 2, 934-937 (1991)
- 34. Rinard G.A., Quinne R.W., Ghim B.T., Eaton S.S., Eaton G.R.: J. Magn. Reson. A 122, 50-57 (1996)
- 35. Rinard G.A., Quinne R.W., Ghim B.T., Eaton S.S., Eaton G.R.: J. Magn. Reson. A 122, 58-63 (1996)
- 36. Tsapin A.I., Hyde J., Froncisz W.: J. Magn. Reson. 100, 484-490 (1992)
- 37. Rinard G.A., Quine R.W., Eaton G.R.: J. Magn. Reson. 144, 85-88 (2000)
- Rinard G.A., Quine R.W., Eaton S.S., Eaton G.R., Froncisz W.: J. Magn. Reson. A 108, 71-81 (1994)
- Blumenfeld L.A., Voevodskkij V.V., Semenov A.G.: Applications of ESR in Chemistry. Novosibirsk: Akad. Nauk SSSR, Sibirsk. Otd. 1972.
- 40. Strandberg M.W.P.: Rev. Sci. Instrum. 43, 307-315 (1972)
- 41. Terman F.E.: Electronic and Radio Engineering, 4th edn., chaps. 3 and 4. London: McGraw-Hill 1995.
- 42. Webber G.D., Riedi P.C.: J. Phys. E Sci. Instrum. 14, 1159-1163 (1981)
- 43. Broekaert P., Jeener J.: J. Magn. Reson. A 113, 60-64 (1995)
- 44. Gueron M.: Magn. Reson. Med. 19, 31-41 (1991)
- 45. Barjat H., Mattiello D.L., Freeman R.: J. Magn. Reson. 136, 114-117 (1999)
- 46. Speight P.A., Jeffrey K.R., Kourtney J.A.: J. Phys. E Sci. Instrum. 7, 801-802 (1974)
- 47. Pandey L., Huges D.G.: J. Magn. Reson. 56, 443-447 (1984)
- 48. Fukushima E., Roeder S.B.W.: J. Magn. Reson. 33, 199-203 (1979)
- 49. Busses M.L., Peterson G.L.: Rev. Sci. Instrum. 49, 1151-1155 (1978)
- 50. Spokas J.J.: Rev. Sci. Instrum. 36, 1436-1439 (1965)
- 51. Hoult D.I.: J. Magn. Reson. 57, 394-403 (1984)

- 52. Viohl I., Gullberg G.T.: J. Magn. Reson. Imaging 4, 627-630 (1994)
- 53. Hoult D.I., Richards R.E.: J. Magn. Reson. 24, 71-82 (1976)
- 54. Reykowski A., Wright S.M., Porter J.R.: Magn. Reson. Med. 33, 848-852 (1995)
- 55. Schnall M.D., Subramanian V.H., Leigh J.S. Jr.: J. Magn. Reson. 67, 129-134 (1986)
- 56. Rinard G.A., Quine R.W., Eaton S.S., Eaton G.R.: J. Magn. Reson. A 105, 137-144 (1993)
- 57. Ernst R.: Adv. Magn. Reson. 2, 1-131 (1966)
- 58. Brunner P., Ernst R.: J. Magn. Reson. 33, 83-106 (1979)
- 59. Konradi M.S.: Rev. Sci. Instrum. 48, 444-448 (1977)
- 60. Traficante D.D.: Concepts Magn. Reson. 2, 63-67 (1990)
- 61. Hoult D.I., Richards R.E.: J. Magn. Reson. 24, 71-85 (1976)
- 62. Hoult D.I., Lauterbur P.C.: J. Magn. Reson. 34, 425-433 (1979)
- 63. Koptioug A.V., Reijerse E.J., Klaassen A.A.K. in: Proceedings of the Joint 29th AMPERE and 13th ISMAR, vol. II, pp. 1144-1145. Berlin, August 2-7, 1998.
- 64. Koptioug A.V., Reijerse E.J., Klaassen A.A.K. in: Proceedings of the 5th National Conference on Physics and Chemistry of Free Radicals, pp. 321-323. Chernogolovka, Russia, 1997.
- 65. Horowitz P., Hill W.: The Art of Electronics, 3rd edn, chap. 7. Cambridge: Cambridge University Press 1990.
- 66. Soliman F.A.S.: Microelectron J. 27, 1-9 (1996)
- 67. Shalsk E.A.: Proc. IRE 50, 212-216 (1960)
- 68. Grivet P.: J. Mol. Struct. 58, 555-576 (1980)
- 69. Armstrong E.H.: Proc. Inst. Radio Eng. 10, 244-260 (1922)
- 70. Frink F.W.: Proc. Inst. Radio Eng. 26, 76-106 (1938)
- 71. Mano K., Hashimoto M.: Z. Naturforsch. A 41, 445-448 (1986)
- 72. Melnick S., Trepanier R.J., Sullivan E.P.A., Whitehead M.A.: J. Mol. Struct. 58, 337-348 (1980)
- 73. Ataka H.: Proc. Inst. Radio Eng. 23, 841-885 (1935)
- 74. Zikumaru Y.: Z. Naturforsch. A 45, 591-594 (1990)
- 75. Suits B.H., Garroway A.N., Miller J.B.: J. Magn. Reson. 132, 54-54 (1998)
- 76. Wu C.-Y., Hsu H.-S.: IEEE J. Solid-State Circuits 31, 614-624 (1996)
- 77. Laber C.A., Gray P.R.: IEEE J. Solid-State Circuits 23, 1370-1378 (1988)
- 78. Blauch A.J., Schiano J.L., Ginsberg M.D.: J. Magn. Reson. 144, 305-315 (2000)
- 79. Ishikawa Y.: MWE'92 Microwave Workshop Digest 1992, 351-356.
- 80. Ishikawa Y., Yamashita S., Hidaka S.: IEEE Trans. Microwave Theory Tech. 41, 2133 (1993)
- 81. Alecci M., Gualtieri G., Sotgiu A.: J. Phys. E Sci. Instrum. 22, 354-359 (1989)
- 82. Harpen M.D.: Magn. Reson. Med. 34(7), 843-849 (1989)
- 83. Van Heteren J.G., Henkelman R.M., Bronskill M.J.: Magn. Reson. Imaging 5, 93-99 (1987)
- 84. Van Heteren J.G., Henkelman R.M., Bronskill M.J.: Magn. Reson. Imaging 5, 101-108 (1987)
- 85. Bogner R.E.: Electron. Eng. 1965, 115-117.
- 86. Schweiger A., Ernst R.R.: J. Magn. Reson. 77, 512-523 (1988)
- Colligani A., Giordano M., Leporini D., Lucchesi M., Martinelli M., Pardi L., Santucci S.: Appl. Magn. Reson. 3, 107-129 (1992)
- Nicholson I., Foster M.A., Robb F.J.L., Hutchison J.M.S., Lurie D.: J. Magn. Reson. B 113, 256– 261 (1996)
- Koptioug A., Nicholson I. in: Proceedings of the 28th Congress AMPERE, pp. 269-270, Canterbury, UK 1996.
- 90. Shane J.J.: Ph.D. thesis, University of Nijmegen, Nijmegen, The Netherlands 1993.
- Koptyug A.V., Reijerse E.J., Klaassen A.A.K. in: Proc. 28th Congress AMPERE, p. 267, Canterbury, UK 1996.
- 92. Koptyug A.V., Reijerse E.J., Klaassen A.A.K.: J. Magn. Reson. 125, 369-371 (1997)
- 93. Wolak J., Hilczer B.: Mol. Phys. Rep. 5, 311-314 (1994)
- 94. Colligani A., Guillon P., Longo I., Martinelli M., Pardi L.: Appl. Magn. Reson. 3, 827-840 (1992)
- 95. Colligani A., Longo I., Martinelli M., Lucchesi M., Pardi L.: Appl. Magn. Reson. 9, 567-579 (1995)

Authors' address: Andrei V. Koptioug, Project JEPP, Mid-Sweden University, Frösö Strand, Öneslingan 5, 831 25 Östersund, Sweden