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Total compensation of pulse transients inside a resonator

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ABSTRACT

The profile of rf pulses that nuclear spins experience inside a resonator deviates from that of rf voltage signals generated by a NMR spectrometer according to users' pulse programming, when change of the profile in time is comparable to or shorter than the time constant of the resonator. In our previous work [Takeda et al., J. Magn. Reson. 197 (2009) 242–244], we proposed active compensation of rf pulse transients, in which the amplitude transient of the rf pulse can be suppressed without sacrificing the Q factor of the probe. Here we extend the idea of active compensation toward total compensation of the amplitude as well as phase transients. By measuring the transient response of the probe to a given excitation using a pickup coil, the response function determining the transient behavior of the probe is numerically obtained. Then, by numerically solving the convolution equation with the help of Laplace transformation, one can obtain the amplitude and phase profiles of the pulse that should be programmed in the spectrometer in order to apply the rf pulses to the nuclear spins as intended. Accurate rf pulsing based on this idea is experimentally demonstrated, and prospect and requirements for coping with the receiver dead-time problem are discussed.

1. Introduction

In pulsed nuclear magnetic resonance (NMR) experiments, nuclear spins placed in a static magnetic field are irradiated with pulsed radiofrequency (rf) magnetic fields. Usually rf irradiation is applied using a resonator or a tank circuit composed of a coil and capacitors, which enhances the efficiency of rf transmission to the spin system by the quality (Q) factor of the resonator. For a typical resonator, the Q factor is on the order of 10^2, while recent progress in cooling the circuit to cryogenic temperatures realized the Q factor exceeding 10^3. According to the theorem of reciprocity [1], the amplitude of the signal induced by the precessing nuclear magnetization is also enhanced by the Q factor of the resonator, leading to much better sensitivity. However, the price to pay for the enhancement of rf irradiation efficiency/signal detection sensitivity is the transient effect of the rf pulses, which increases with the Q factor of the resonator. In particular, the profile of the pulsed magnetic fields that the nuclear spins experience inside the resonator deviates from that of the intended one at the leading and trailing edges of the pulse. The transient effects last for a time interval $\tau \sim 2Q/\omega_0$, where ω_0 is the angular resonant frequency of the tank circuit. For the current typical NMR experiments done with $Q \sim 100$ and $\omega_0/2\pi \sim 100$ MHz, τ is on the order of several hundred of nanoseconds, while τ can be several microseconds for NMR studies of exotic low-γ nuclei.

In addition to profile deviation, phase deviation at the pulse edges can also happen when, in particular, the excitation frequency is off-resonance from the tuned frequency. The phase transient causes an instantaneous frequency chirping, and thereby an additional time-varying z-component of the effective magnetic field in the rotating frame. These phenomena were investigated in detail based on a circuit equation [2,3].

The rf pulse transients result in cumulative errors in multiple pulse experiments [4–7]. Even though the effect of the transient-induced pulse imperfection has been extensively studied [2,8] and a number of error-tolerant pulse sequences and ways of spectrometer tuning have been proposed [2,9–13], accurate rf pulsing leading to precise control of nuclear spin systems would still be desirable in order to explore new possibility of magnetic resonance spectroscopy, including sophisticated multiple pulse experiments, very short pulses with strong rf irradiation using a micro-coil, pulsed electron spin resonance (ESR) spectroscopy, application to quantum computing [14], and so on.

In order to suppress the amplitude transient, a phase inverted-pulse was proposed by Hoult [15]. Very recently, we have demonstrated an approach for calculating the excitation rf voltage profile back from the intended rf filed profile based on linear response of the probe resonant circuit. And also, we have experimentally demonstrated that the amplitude transients can be successfully

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compensated [16]. However, the phase transient has not been dealt with yet.

In this paper, we extend the idea of active compensation to totally suppress the amplitude and phase transients. By employing a field pickup coil, the transient response of the NMR circuit is measured to characterize the circuit. The response function of the circuit, obtained numerically from the response within the scope of linear response theory, is then used for calculating the amplitude and phase of the excitation pulse resulting in an intended pulse shape inside the sample coil. This strategy may remind readers of the ideas of feedback of nuclear magnetization to the tank circuit, such as radiation damping [17] and its compensation [18–20], automatic selective excitation [21], self-sustained maser oscillation [22], non-linear spin dynamics [23], chaotic spin behavior termed as spin turbulence [24], and image-contrast enhancement in MRI [25]. They are, however, distinct from the strategy of total compensation presented here, in the sense that the former rely on concurrent feedback while the latter lets us prepare the way that we design the excitation pulse in advance.

The idea of total compensation presented here would also be of considerable interest from the viewpoint of coping with the problem of receiver dead-time. The NMR receiver is usually protected from high-power rf pulses by a duplexer, which blocks high-voltage signals but not perfectly. Since the amplitude of the leaked rf pulses is still orders of magnitude larger than the NMR signals, data acquisition is not possible until the transient tail of the rf pulses has decayed completely. This time interval between the trailing edge of the pulse and the start of signal acquisition is called as the receiver dead-time. The dead-time problem is serious when the resonance line is so broad that the NMR signal decays before data acquisition. Even though the decay may be recovered by echo techniques, this is only possible when the decay mechanism is coherent, and there is no way of recovering decoherence usually characterized by a time constant T_2. Possibility and prospect for dead-time reduction are also discussed in this work.

2. Basic concept

In this work we restrict our discussion to a linear system by assuming a linear relation between an excitation $v(t)$ programmed in a spectrometer and the magnetic field $B_1(t)$ generated in a resonator. This is valid for most NMR probes composed of inductors and capacitors. According to linear response theory, their relation can be expressed using a convolution as

$$B_1(t) = h(t) * v(t) = \int_{-\infty}^{t} h(\tau - t)v(\tau)\,d\tau, \tag{1}$$

where $h(t)$ is a response function. In the present case, $h(t)$ is the field profile created by an impulse excitation. In order to back-calculate the voltage profile $v(t)$ that gives a desired field profile $B_1(t)$ inside the resonator, we apply Laplace transformation, defined as $\mathscr{L}[a(t)] = A(s) = \int_0^\infty a(t)\exp(-st)\,dt$, to Eq. (1). We then obtain

$$B_1(s) = H(s)V(s), \tag{2}$$

where $B_1(s) = \mathscr{L}[B_1(t)], H(s) = \mathscr{L}[h(t)]$ and $V(s) = \mathscr{L}[v(t)]$. Note that Laplace transformation of a convolution results in a product of the individual Laplace transformed functions. Then, $V(s) = B_1(s)/H(s)$, and we obtain $v(t)$ by performing inverse Laplace transformation as

$$v(t) = \mathscr{L}^{-1}\left[\frac{B_1(s)}{H(s)}\right]. \tag{3}$$

We call $v(t)$ obtained in this way as the "*compensation pulse.*"

In our previous work, $h(t)$ was approximated by an exponential function as $h(t) = \exp(-\omega_0 t/2Q)$, and an analytical solution $v(t) = B(t) + \frac{2Q}{\omega_0}\frac{d}{dt}B(t)$ was obtained, which was shown to work well for compensation of the amplitude transient arising in the probe [16]. In this work, we propose to compensate the amplitude and phase transients arising from the *net* system including a power amplifier, transmission lines, filters, as well as the resonator. For this purpose, a field pickup coil is employed and placed beside the NMR sample coil as schematically described in Fig. 1, so that a small portion of the rf magnetic field produced by the sample coil can be monitored. The voltage response $y(t)$ induced by a given excitation $u(t)$ is represented as

$$y(t) = h_{\mathrm{M}}(t) * h(t) * u(t), \tag{4}$$

where $h_{\mathrm{M}}(t)$ is the response function of the pickup coil. For a pickup coil with an inductance L and an input impedance R of an oscilloscope, the response function $h_{\mathrm{M}}(t)$ is analytically given as $h_{\mathrm{M}}(t) \propto \delta(t) - (R/L)\exp(-Rt/L)$, where $\delta(t)$ is the Dirac delta function. Here we choose such L and R that the time constant (L/R) is much shorter than the time scale we deal with, and thereby make an approximation $h_M(t) \sim \delta(t)$. Then, Eq. (4) becomes

$$y(t) \sim h(t) * u(t). \tag{5}$$

Now we apply Laplace transformation to Eq. (5) as

$$Y(s) = H(s)U(s), \tag{6}$$

where $Y(s) = \mathscr{L}[y(t)], U(s) = \mathscr{L}[u(t)]$. Using Eq. (5), the response function represented in the Laplace domain can be obtained from measured response $y(t)$ to the excitation $u(t)$>

$$H(s) = \frac{Y(s)}{U(s)}. \tag{7}$$

Substituting Eq. (7) into Eq. (3), we arrive at

$$v(t) = \mathscr{L}^{-1}\left[\frac{B_1(s)U(s)}{Y(s)}\right]. \tag{8}$$

Thus, once the response $y(t)$ to a certain excitation $u(t)$ has been measured, the shape (including amplitude as well as phase) of the compensation pulse can be calculated for any realistic pulse shapes $B_1(t)$ that we intend to apply to the spin system inside the resonator. Here we exclude those "unrealistic" pulse shapes which have frequency components spanning outside the bandwidth of the resonator from our discussion, for such components are out of reach of the compensation approach. An example of such is a rectangular pulse, whose power spectrum is plotted in Fig. 2a together with the resonance curve of a tank circuit resonating at 12.7 MHz with Q = 35. Even though the rectangular pulses are the most frequently used in NMR pulse sequences, they cannot really be perfectly rectangular, because the actual pulse cannot rise and fall at an infinite rate. Instead, we deal with something similar but with a moderate bandwidth by considering a pulse with smooth rising and trailing edges represented by a cosine function, defined as

$$B_1(t) = \begin{cases} \frac{1}{2}[1 - \cos(\pi t/d)]\sin(\omega_0 t + \phi) & (0 \leqslant t < d), \\ \sin(\omega_0 t + \phi) & (d \leqslant t < T), \\ \frac{1}{2}[1 - \cos(\pi(T + d - t)/d)]\sin(\omega_0 t + \phi) & (T \leqslant t < T + d), \\ 0 & (\text{otherwise}), \end{cases} \tag{9}$$

where T and d represent the plateau interval and the transition periods, respectively. Fig. 2b shows a power spectrum of the cosine pulse that just fits in the resonance curve of the tank circuit.

On the other hand, the frequency bandwidth of the probe excitation $u(t)$ needs to be wide enough to cover that of the resonator in order to extract the response of the resonator. One of such options that we used in the present study is a step sinusoidal function with a carrier frequency ω_0, given by

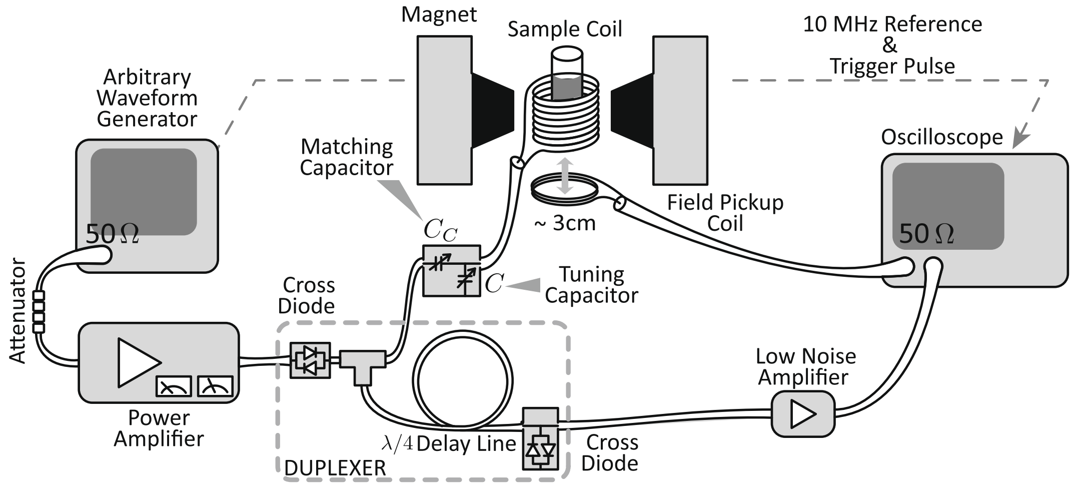

Fig. 1. A schematic diagram of the experimental setup.

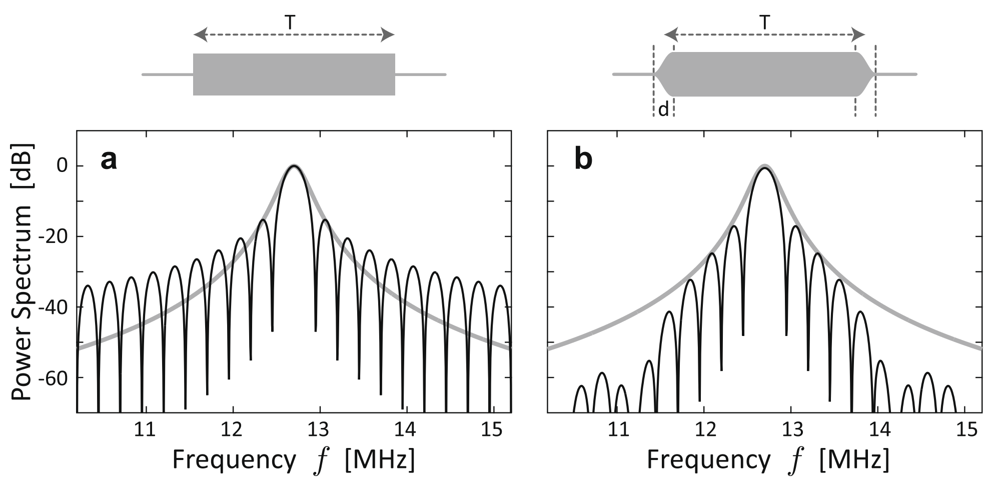

Fig. 2. (a) A normalized spectrum of a rectangular pulse with a duration T of 4 μs (solid line) and a resonance curve of a resonator with a Q factor of 34 and resonant frequency $\omega_0/2\pi$ of 12.7 MHz (gray line). (b) A normalized spectrum of a cosine pulse with a duration T of 4 μs and transition periods d of 1 μs (solid line).

$$u(t) = \begin{cases} \sin(\omega_0 t) & (t > 0), \\ 0 & (t < 0). \end{cases} \tag{10}$$

3. Experimental

A schematic diagram of the experimental setup is shown in Fig. 1. An rf pulse was created by an arbitrary waveform generator (AWG7102, Tektronix), and amplified with a 500 W solid-state amplifier (N146-5759C, THAMWAY). Two cross diodes and a quarter-wavelength delay line compose a duplexer; the amplified pulse was transmitted into the resonator and an NMR signal, which was exceedingly small compared with that of the rf pulse, was lead to a low-noise preamplifier and acquired with an oscilloscope (DPO7254, Tektronix). Signal acquisition was triggered by the AWG. Both the clocks of the oscilloscope and the AWG were synchronized using a reference clock at 10 MHz.

The resonator was composed of a solenoid coil (sample coil) with an inductance of 0.3 μH and a diameter of 4 mm, variable tuning and matching capacitors. Its resonance frequency $\omega_0/2\pi$ was adjusted to 12.7 MHz. The quality factor, Q, of the resonator was measured to be 35 using a network analyzer. In order to monitor the magnetic field, a field pick up coil with a diameter of 7 mm, number of turns of 3, and an inductance of 80 nH was placed 3 cm apart from the sample coil. The coupling between the sample coil and the pickup coil was measured to be −33 dB. Since only a tiny fraction (less than 0.1%) of the applied rf power is monitored, the presence of the pickup coil had little effect on the performance of the tank circuit. We did not find any appreciable extra noise caused by the pickup coil, either. The signal of the monitored rf pulse was stored on the digital oscilloscope at a sampling rate of 10 GHz, and digitally demodulated with respect to the carrier reference signal into the in-phase and quadrature components.

4. Results and discussions

Fig. 3a and b shows the step sinusoidal excitation $u(t)$ given in Eq. (10) and its rotating frame representation with respect to its carrier frequency of 12.7 MHz. The response $y(t)$ of the resonant circuit to $u(t)$ measured using the pickup coil and its in-phase and quadrature components are shown in Fig. 3c and d. Then $u(t)$ and $y(t)$ were numerically Laplace transformed into $U(s)$ and $Y(s)$.

Firstly, the transient effect without compensation was examined for the cosine input pulse with a rise/fall time interval d of 1 μs. This input pulse, plotted in Fig. 4a and in c using the quadrature representation, resulted in a distorted rf pulse profile with the transient effect, as shown in Fig. 4e.

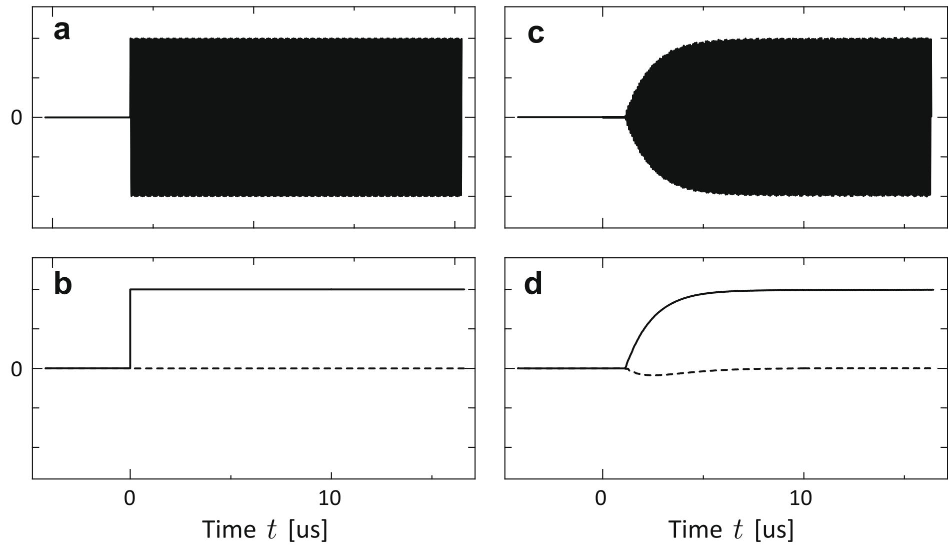

Fig. 3. (a) A step sinusoidal function for probe excitation and (c) its response. (b) and (d) The quadrature representations of (a) and (c) with respect to the carrier frequency of 12.7 MHz. Solid and dashed lines denote in-phase and quadrature phase components, respectively.

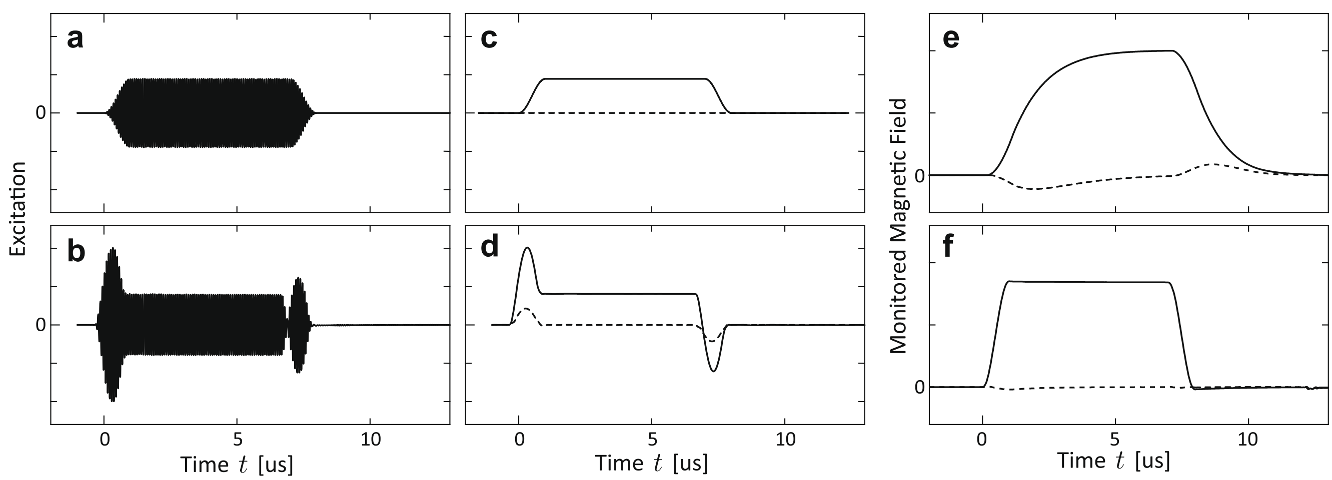

Fig. 4. (a) A cosine pulse with a transition time d of 1 μs and a width T of 7 μs and (b) the corresponding compensation pulse. They are represented in terms of the two orthogonal phases in (c) and (d). (e) and (f) Monitored fields for the excitations (a) and (b). Solid and dashed lines denote in-phase and quadrature phase components, respectively.

Next, this cosine pulse shape $B_1(t)$ was Laplace-transformed into $B_1(s)$, and using $U(s)$ and $Y(s)$, the shape of the corresponding compensation pulse was calculated according to Eq. (8). Fig. 4b and d shows the computed compensation pulse and its in-phase and quadrature phase components. The resultant magnetic fields shown in Fig. 4e and f compensated both the amplitude and phase transients, giving the intended cosine pulse shape inside the NMR coil. The effect of the characteristic shape of the compensation pulse in Fig. 4d at the edges is to cancel the pulse shape deformation arising from the transient effect.

The effect of the phase transient in the pulse is to tip the spin system about an axis perpendicular to the intended axis unintentionally. This effect is revealed prominently for $k\pi$ pulses with $k = 1, 2, \cdots$, as one observes a dispersive NMR signal that would not appear if it had not been for the phase transient. Fig. 5a shows ^{1}H nutation spectra of water measured with uncompensated cosine pulse for various pulse widths. As seen here, the spectra for pulse widths of 4.2 and 8.4 μs corresponding to the π and 2π pulses were dispersive. As shown in Fig. 5a, the phase transients at the leading and the trailing edges have the opposite sign. Thus, when the nutation angle is close to 2π, the excess tilt of the spins caused by the phase transient at the beginning of the pulse tends to cancel that at the end of the pulse, while they reinforce with each other for the nutation angle close to π. For this reason the amplitude of the dispersive signal for the π pulse is larger than that for the 2π pulse.

The advantageous feature of the compensation pulse is demonstrated in Fig. 5b, where the nutation spectra became purely absorptive, indicating that the intended, phase transient-free magnetic field pulse was produced inside the resonator. As seen here, the spectra for pulse widths of 4.2 and 8.4 μs were close to ideal with much less transient effect.

Without compensation, the transient at the edges of the pulse approximately shows an exponential behavior with a time constant τ given by the Q factor and the resonance frequency ω_0 as $2Q/\omega_0$ [16]. In the present case, $\omega_0/2\pi$ and Q were 12.7 MHz and 35, so that $\tau \sim 0.88$ μs. Since the time required for the pulse to get full amplitude is given by $\sim 5\tau$, the shortest possible pulse would be limited to ~ 4 μs. For a pulse with a shorter width than that, the amplitude would begin to fall down before reaching the

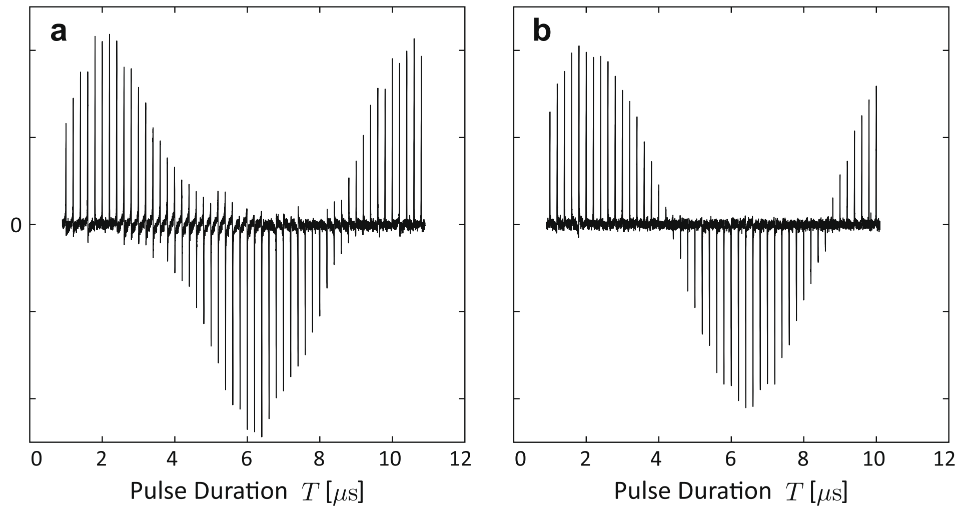

Fig. 5. ^{1}H nutation spectra of water using cosine pulses (a) without compensation and (b) with compensation. The static magnetic field was 298 mT, and the resonance frequency was 12.7 MHz.

maximum. The time constant τ would become shorter under high magnetic field, indicating that transient effects are decreased. For example, the time constant τ of a resonant circuit tuned to the frequency of 500 MHz and having a Q factor of 120 becomes 76 ns, while the time constant of a resonator for low-γ, e.g. $\omega_0/2\pi =$ 2 MHz and $Q = 20$, becomes 3.18 µs. This evaluation shows that problems in terms of transients and receiver dead-time are serious for low frequency resonators. As for pulsed-ESR spectroscopy, the time constant of a typical X-band resonator ($Q \sim 1000$) is 35 ns, which is comparable to $\pi/2$ pulse in typical ESR and requiring echo technique to avoid receiver dead-time.

On the other hand, the compensated cosine pulse with the transition time d of 1 µs has successfully been demonstrated in this work, making much shorter and transient-free pulses feasible. The idea of transient compensation proposed here would be of interest for multiple pulse experiments, where the cumulative effect of the transients in the individual pulses can seriously disturb the spin dynamics. The effect of the transients in multiple pulse sequences has been extensively discussed in the literature with a practical tip of de-tuning the probe for reducing the phase transient [12,26]. In contrast, the present work provides a promising alternative approach based on the protocol as described above, requiring neither probe de-tuning nor reduction of the Q factor. Thus, the performance of the latest high-Q NMR probes with a cryogenically cooled coil can be fully exploited. In particular, the high-Q probes for solid-state NMR [27] would need compensation in order to apply intense and thereby short rf pulses for manipulating the relatively strong spin interactions that are present in the solid-state. Since the principle of the present approach is also valid for microwave resonators, the idea of total compensation would also find interest in pulsed-ESR, considering recent progress in signal generation technology at up to several tens of gigahertz. In addition, unitary evolution of the spin system under accurate pulsing would be promising in quantum computing using nuclear/electron spins.

In order to examine the prospect for reducing the receiver dead-time, the signal voltages at the receiver input were compared for the uncompensated and compensated cosine pulses. As shown in Fig. 6a, the tail of the uncompensated pulse lasted for ca. 9 µs before the voltage dropped by six orders of magnitude, giving a dead-time of 13 µs. On the other hand, the tail of the compensated pulse dropped more rapidly to one thousandth the initial value within the transition time d of 1 µs, as demonstrated in Fig. 6b. After that, however, the residual tiny signal trailed at a rate nearly equal to that in the case of the uncompensated pulse. In this case, the dead-time was reduced to 9 µs. Such a slight improvement was ascribed to the residual transient tail that failed to be compensated. This result suggests that the compensation should be implemented far more accurately for further dead-time reduction. In this context, we find room for improvement regarding to the following two respects.

First, it is necessary to deal with non-linear effects. Even though the present approach relies on the linearity of the system, the practical NMR system uses the duplexer, which shows non-linear behavior when the amplitude of the signal is on the order of or smaller than the threshold level of the cross diodes. Also, the power amplifier can be non-linear. An extended total compensation approach which works even in the presence of non-linear components could be developed by applying the idea of non-linear models, which have been already proposed in communication engineering. Then, compensation of the transient tail would be much closer to ideal.

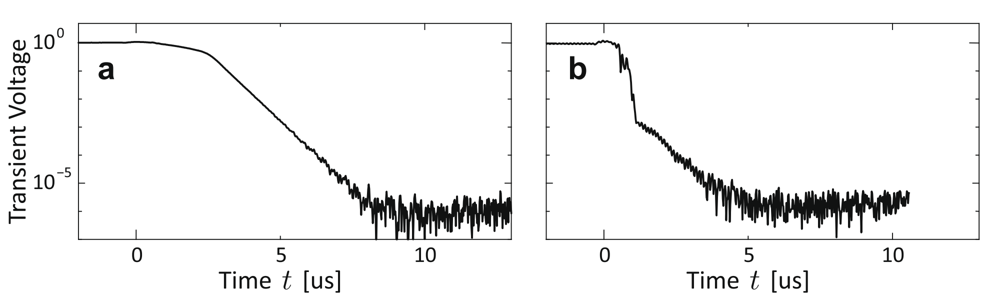

Fig. 6. Decay of the transient signals after pulsing at the receiver (a) without compensation and (b) with compensation.

What we need to consider then is the accuracy of measurement of the step response $y(t)$, and that of conversion of numerically derived compensated pulse shape $v(t)$ into the analog voltage signal. They are both determined by the dynamic range and sampling rate of A/D or D/A conversion. In the present study the dynamic range and the sampling rate for measurement of $y(t)$ were 2^8 and 10 GS/s, while those for signal generation according to $v(t)$ were 2^{10} and 10 GS/s, respectively. Currently studies on practical requirement for the accuracy of response measurement and signal generation are in progress, in the hope of suggesting the desirable specification of future NMR spectrometers capable of high-performance total compensation of rf pulse transients.

5. Conclusion

The way in which the transient effects appear is determined by the response function specific to the electronic circuit. Once the response function has been obtained by measuring the response of the resonator to a certain excitation using the pickup coil, it is possible to design in advance such rf pulse shape that results in the intended shape inside the resonator, as described in this work. Since the response function obtained numerically in the form of a complex function characterizes both the amplitude and phase responses of the resonator, total compensation of the amplitude and phase transients has become feasible, in contrast to the previous reports dealing with only the amplitude transient.

Here we have demonstrated total compensation of both the amplitude and phase transients using the cosine pulse, and explored possibility for suppressing the unwelcome outcomes we encounter without compensation, including the dispersive spectrum in a single pulse experiment and the receiver dead-time problem. The latter issue was found to be more demanding, putting stronger requirements regarding to the accuracy in measurement of the response and in generation of the compensation pulse, as well as the effect arising from the non-linear devices in the system.

It would be worth emphasizing here that the response of the probe alone can differ from that of the *whole circuit* composed of the power amplifier, transmission lines, filters, etc. as well as the probe. It follows that the response function of the net circuit should be derived whenever the user makes change to the circuit assembly. Since the pickup coil can be placed without any serious disturbance to the original tank circuit, it would be desirable if every standard commercial probe were equipped with a pickup coil for measuring the response of the circuit, and a commercial spectrometer were capable of numerically deriving the response function after auto tuning, and automatically generating the pulse shape that results in the intended one inside the NMR coil. Then, the transient compensation approach proposed here would find a number of applications, including liquid-state NMR using cryoprobes, multiple-pulse solid-state NMR spectroscopy which is currently carried out using *Q*-damped probes, NMR measurements on exotic low-γ nuclei, and so on.

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