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A new pulse width reduction technique for pulsed electron paramagnetic resonance spectroscopy

Yasunori Ohba [a,d,*], Shigeaki Nakazawa [b,d], Shunji Kazama [c,d], Yukio Mizuta [c,d]

[a] *Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Katahira 2-1-1, Aobaku, Sendai 980-8577, Japan*
[b] *Department of Chemistry, Graduate School of Science, Osaka City University, 3-3-138 Sugimoto, Sumiyoshi, Osaka 558-8585, Japan*
[c] *JEOL Ltd., 3-1-2 Musashino, Akishima, Tokyo 196-8558, Japan*
[d] *CREST, JST, Kawaguchi 332-0012, Japan*

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Abstract

We present a new technique for a microwave pulse modulator that generates a short microwave pulse of approximately 1 ns for use in an electron paramagnetic resonance (EPR) spectrometer. A quadruple-frequency multiplier that generates a signal of 16–20 GHz from an input of 4–5 GHz was employed to reduce the rise and fall times of the pulse prepared by a PIN diode switch. We examined the transient response characteristics of a commercial frequency multiplier and found that the device can function as a multiplier for pulsed signal even though it was designed for continuous wave operation. We applied the technique to a Ku band pulsed EPR spectrometer and successfully observed a spin echo signal with a broad excitation bandwidth of approximately 1.6 mT using 80° pulses of 1.5 ns.

Keywords: Pulsed EPR; Frequency multiplier; PIN diode switch; Excitation bandwidth

1. Introduction

Pulsed electron paramagnetic resonance (EPR) is a powerful method for investigating the structure, electronic state, and dynamics of paramagnetic molecules in both the steady and transient states [1,2]. In some of these applications, such as Fourier transform (FT) EPR [2] and double quantum coherence (DQC) distance measurements [3], the microwave pulse must be narrow and intense to obtain a large excitation bandwidth. For example, to obtain the highest sensitivity in FT-EPR utilizing the multi-channel advantage, an entire EPR spectrum must be excited by a single pulse; a bandwidth of more than 10 mT is needed for the excitation of organic free radicals, especially those in a rigid matrix. In the case of DQC measurements, simultaneous excitation of two interacting electron spins is essential, meaning that the bandwidth must cover the entire spectra of both spins. Thus, to be useful for most free radical systems, the pulsed magnetic field must be greater than 5 mT; correspondingly, the pulse width must be less than 1.5 ns. If we consider multiple pulse experiment, shorter and stronger pulses are needed. To the best of our knowledge, the largest excitation width is obtained using a single pulse of 2.5 ns [4]. We think that further reduction of the pulse width is important to increase the excitation bandwidth.

Until now, the excitation bandwidth has been limited by the minimum pulse width determined by the switching and transition (rise and fall) times of commercially available microwave PIN diode switches. The switching time is defined as the delay time to microwave output from the control signal and is approximately 5–7 ns for a fast PIN diode switch. This time is also related to the minimum pulse width, though this parameter is generally not specified. The rise (or fall) time is defined as the time taken

* Corresponding author. Address: Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Katahira 2-1-1, Aobaku, Sendai 980-8577, Japan. Fax: +81 22 217 5616.
E-mail address: yohba@tagen.tohoku.ac.jp (Y. Ohba).

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for the microwave amplitude to change from 10% (or 90%) to 90% (or 10%), and is approximately 2 ns for the fastest version. These parameters depend not only on the characteristics of the diode but also on the biasing circuit (termed the driver), and are strongly related to other properties such as over/under shoot, video feed-through, isolation, and insertion loss. Compromise among these parameters limits the present speed of PIN diode switches, meaning that demand exists for more sophisticated design and fabrication of both the driver and microwave circuitry. As in the case of Ref. [5] where a single pulse of 2 ns was realized, one need custom made switch of better performance.

A pulse of approximately 1 ns has been used in pulse radar reflectometers in nuclear plasma diagnosis [6,7], but specially constructed switching devices are used in this application, resulting in limited availability. A short and intense microwave pulse is also used in linear accelerators; several pulse compression techniques have been developed for this purpose [8,9]. These techniques are attractive because they not only enable the microwave pulse to be shortened, but also intensified by several orders of magnitude as high as megawatts; however, pulsed EPR experiments require numerous series of pulses with accurate inter-pulse distances, phase modulation, and well-defined coherency. This is very difficult to achieve using existing pulse compression techniques.

In the present paper, we propose a new technique that reduces the transition times of the microwave pulse by utilizing a nonlinear property of a frequency multiplier that enables the generation of short microwave pulses of approximately 1 ns. A frequency multiplication technique was developed following the recent increase in frequency bandwidth in various applications, such as in computing and digital communications, that require a very stable frequency source as high as 100 GHz or more [10,11].

One of the best current methods for obtaining a stable and highly coherent signal is the frequency multiplication of a stable frequency source of lower frequency, because the construction of such a high-frequency oscillator is very difficult. In these techniques, a desired frequency component is selected from the harmonics that result from the nonlinearity of devices such as diodes or amplifiers. Our basic concept is that the nth harmonic signal is included in n time products of the input signal; therefore, if the input signal is expressed as a product of continuous wave (cw) and envelope functions, the envelope function should also be multiplied in the output harmonics. Thus, if we write the input signal as $S_I(\omega) = f(t)\cos(\omega t)$ by writing the envelope function as $f(t)$, the envelope of nth harmonics of the frequency $n\omega$ should be expressed by n multiples of the envelope function as $f(t)^n$.

It is clear that the time of the transient part of any function is shortened by multiplication. As a simple example, considering a Gaussian pulse with characteristic width τ, the envelope function is expressed as $\exp(-t^2/\tau^2)$. The n time product of the envelope function becomes $\exp(-nt^2/\tau^2)$ and its width is $n^{1/2}$ times narrower than that of the input signal. To the best of our knowledge, only one report describes a spectrometer that uses a high-frequency pulse obtained by a frequency multiplication technique [12,17]. The authors of this earlier paper stated that the advantage of this technique lies in improvements in the isolation of the PIN switch, which ascribes to the same principle as in our technique; that is, enhancement of the amplitude ratio by multiplication.

This effect can be expected for any envelope function having a finite length of transient parts, although a general numerical expression of width reduction cannot be obtained because the expression of the rise or fall time depends on the explicit functional shape. The actual multiplication number for an envelope function is not easily obtained because the operation principle of an actual device is complicated, and it is difficult to calculate the input power dependence of the amplitude of a specific harmonic signal [13].

To test the method, we used the Ku band (17–18 GHz) as the EPR frequency and a multiplication number of 4 to demonstrate the reduction of the pulse width by the frequency multiplier. This choice was made for practical reasons; i.e., the availability of commercial devices. Thus, we used a MARKI DAD0405 frequency quadruple multiplier that consists of two doublers and an amplifier. The DAD0405 is designed for input and output frequencies of 4–5 GHz and 16–20 GHz, respectively, and input and output levels of 14–8 dBm and 3 dBm, respectively.

Very recently, Bolton et al. published a work on the multiplication technique for sub-nanosecond pulse generation at 94 GHz and showed an application to high resolution time-domain reflectometry suggesting usefulness of the technique in other applications including pulsed EPR [14]. Present our work is concerned with detailed analysis of the pulse operation of a multiplier and present a practical application to pulsed EPR.

2. Results

2.1. Nonlinear characteristics of the commercial quadruple-frequency multiplier DAD0405

The bandwidth of a multiplier is important in our application because the pulsed signal contains a wide range of frequency components. We measured the output power as a function of input frequency from 3.8 GHz to 5.2 GHz at 10 dBm input level, as shown in Fig. 1. The output power was approximately 0 dBm and almost constant (±1.4 dBm) over the frequency range 4.0–5.0 GHz. For the present application, we needed the bandwidth of 0.13 GHz, which corresponded to the shortest rise and fall time (approximately 2 ns) limited by the PIN diode switch operating at 4–5 GHz. Thus, roughly speaking the bandwidth of DAD0405 was sufficient for pulse operation in the frequency range 4.2–4.8 GHz. Although Fig. 1 indicates that DAD0405 can be used over the full range of 4.0–5.0 GHz, we avoided the bandwidth edges because out-

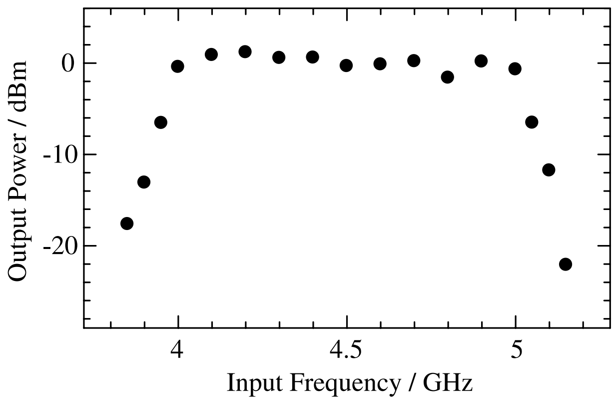

Fig. 1. Frequency characteristics of a DAD0405 quadruple-frequency multiplier. A cw signal of 10 dBm from an Agilent N530A network analyzer was fed into the DAD0405. The output power and frequency of the DAD0405 were measured using an Agilent 53150 A frequency counter that could also measure power. The output signal of the DAD0405 was filtered by a band-pass filter (16–20 GHz) to remove unwanted harmonics.

put power rapidly decreases above and below 5.0 GHz and 4.0 GHz, respectively.

Input power dependence is important for pulse operation because the multiplication number for the envelope function is determined by nonlinearity in this behavior. Therefore, the output power was measured as a function of the input power for several frequencies, as shown in Fig. 2. For power input larger than 8 dBm, output power was almost independent of input power; in contrast, for input power lower than 4 dBm, output power depends almost linearly on input power in the logarithmic plot. The slope value of the plot was calculated to be 4.1 ± 0.1 for input power less than 4 dBm in the frequency range 4.2–4.8 GHz; this value suggests that the multiplication number for the envelope function is approximately 4. It must be noted that the slope is less than 4 for frequencies less than 4.2 GHz and greater than 4.8 GHz. This provides another reason why the input frequency must be limited to the range 4.2–4.8 GHz.

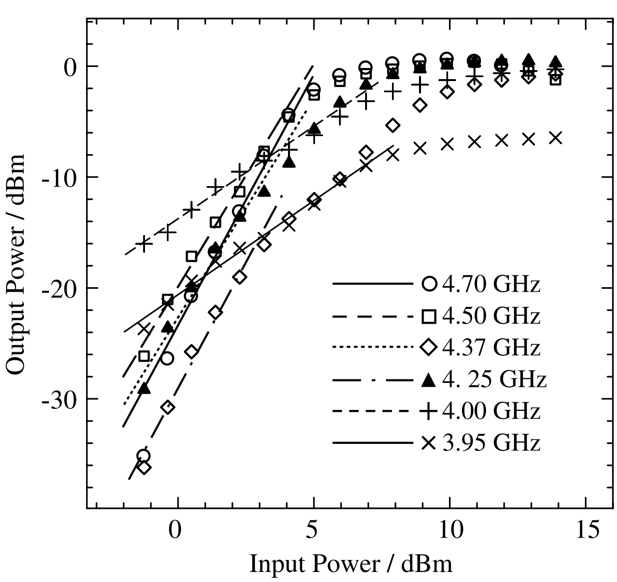

Fig. 2. Power dependence of the output power of the DAD0405 frequency multiplier. The symbols represent the observed data, and the lines are fitted to the linear part (input power below 2 dBm) of the data. The experimental details are the same as for Fig. 1.

2.2. Pulse response of the quadruple-frequency multiplier

The above results indicate the suitability of DAD0405 in pulse operation. To proceed to practical application, we examined the response of DAD0405 to a pulsed input signal by measuring the peak power of the output pulse. An input pulse of width 10 ns was produced by switching a cw signal of 4–5 GHz using a PIN diode switch. The shape of the input pulse was maintained by controlling the power of the input pulse by attenuation after switching by the PIN diode rather than changing the cw input power, because pulse shape is affected by cw power. The peak power of the pulsed signal was measured using a calibrated diode detector for both the input and output pulses.

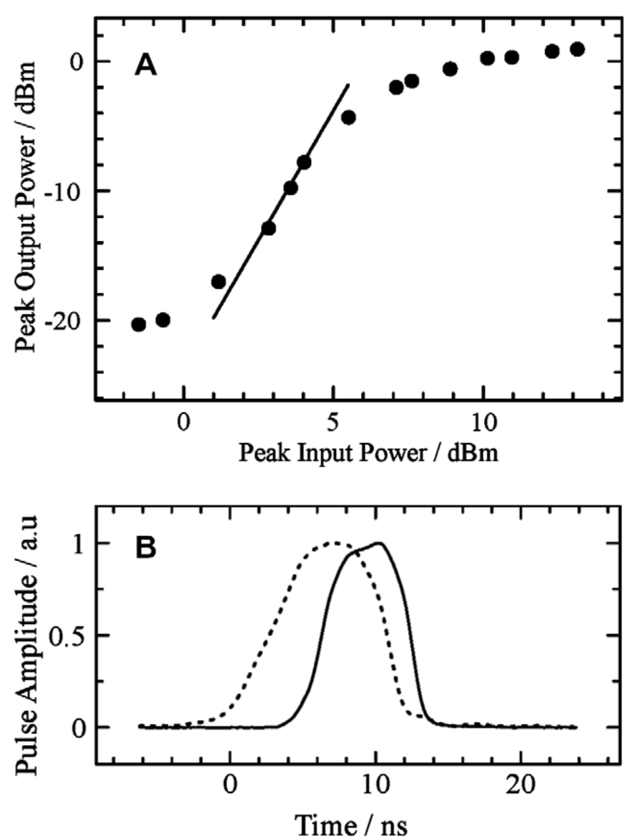

Fig. 3. Power dependence (A) and waveform (B) of the pulsed input and output signals of DAD0405. The circles represent peak power of the output pulse signal with line fitting for a slope value of 4.0 (A); the solid and dotted lines represent the input and output signals, respectively, for power dependence (B). The carrier frequencies of the input and output pulses were 4.5 GHz and 18 GHz, respectively. The power of the microwave pulse was measured using a calibrated diode detector, Krytar model 303BK or 703BK, terminated directly into a 50Ω load of a LeCroy WavePro7300 3 GHz digital sampling scope. The output signal of the DAD0405 was filtered to remove unwanted harmonics using a band-pass filter (16–20 GHz) placed at the input of the diode detector.

The observed input power dependence was similar to that of a cw signal, as shown in Fig. 3A. At input power greater than 10 dBm, output power was independent of input power; for input power less than 10 dBm, output power was almost proportional to input power. Again, the obtained slope value was approximately 4 (solid line in Fig. 3A in the logarithmic plot); this confirms that quadruple multiplication also works for the pulsed signal.

Fig. 3B shows the pulse shape of input and output signals for an input power of 6 dBm, which is considered to be the optimum power for nonlinear reduction of the transition time. The rise time of the input pulse, 4.8 ns, is reduced to 3.1 ns in the output pulse. Taking the time for 10–90% change in amplitude as the rise time, the expected rise time for the multiplied pulse was calculated as the time for 56% ($0.1^{-1/4}$) to 94% ($0.9^{-1/4}$) transition of rise of the input pulse by applying the multiplication number 4. Using this method, the expected rise time was calculated to be 2.3 ns, which is close to the observed value. The fall time was also reduced from 3.0 ns to 2.1 ns; the reduced value was close to the 1.9 ns calculated for 56–94% transition of the 4 GHz pulse. Reduction was somewhat smaller than that expected for simple multiplication for both transition times. This discrepancy occurs because the multiplication number is not constant over the power range through which a signal rises or falls, and because the number 4 used in the present study is the maximum possible value, as is shown in Fig. 3A. The optimum input level of 6 dBm is significantly lower than that specified for cw operation. For the higher input level, we observed distortion and elongation in the output pulse. This may suggest the existence of some kind of saturation effect in DAD0405.

2.3. Application to a homodyne EPR system

The present technique was tested on a Ku band (17.5 GHz) pulsed EPR. Because our spectrometer is designed for homodyne detection, the Ku band source signal must be converted to the 4 GHz region using a 1/4 frequency divider, as shown in the block diagram in Fig. 4. To produce a pulse shorter than 10 ns at 4 GHz, we employed two PIN diode switches connected in series; each switch was driven by a TTL gate signal of the same width (20 ns) but shifted 0–20 ns in time. Use of the overlapped part of the two switches produced a narrow pulse of approximately 3 ns. The pulse width was controlled by the delay time between the two TTL signals. The 4 GHz pulses were multiplied, brought into the frequency of the spectrometer, and then returned to the EPR spectrometer.

Fig. 5A shows the 4.4 GHz and multiplied 17.6 GHz pulses, clearly demonstrating a reduction in pulse width from 2.2 ns to 1.5 ns in full-width at half-height. There is also significant difference in shape between the input and output pulses: the 4.4 GHz pulse has significant tailing of the rising and falling edges, but the shape of the output pulse closely approximates a single Gaussian function. The existence of this tailing suggests the limitations of the technique in producing a narrow pulse using two PIN diode switches. Reducing the overlapping output region of the two switches also reduces the peak amplitude because the rise is not fast enough, while the tailing parts remain constant. Thus, the relative amplitude of the tailing parts with respect to the peak amplitude increases with decreasing pulse width. Frequency multiplication improved the pulse shape to more closely approximate a Gaussian

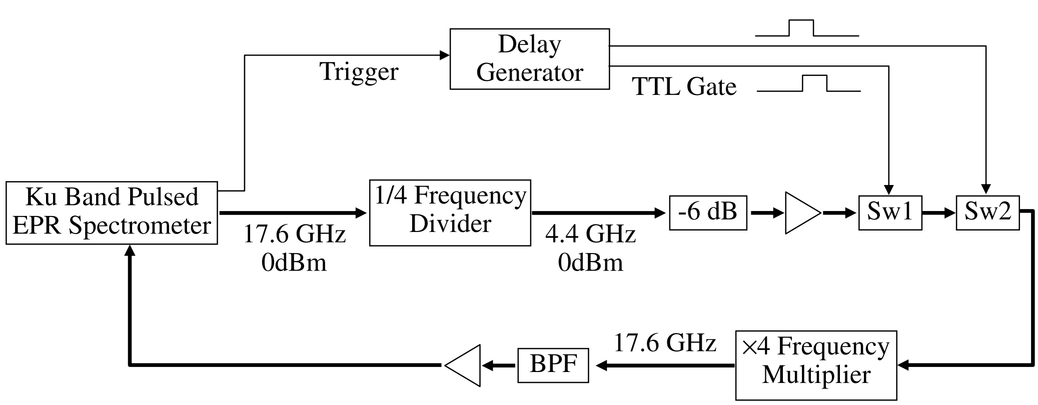

Fig. 4. Block diagram for measurement of the two-pulse spin echo. The thick arrows represent microwave signal and the thin arrows represent TTL pulse signals. We used a CENTELLAX UXC 20PE 1/4 frequency divider, MARKI A0120 amplifiers, MITEQ S147DU1M PIN diode switches (SW1 and SW2), MARKI DAD0405 4× frequency multiplier, a 16–18 GHz band pass filter (BPF), and a Stanford DG535 delay generator. A Ku band EPR spectrometer was constructed based on a JEOL FA series pulsed EPR system. The 16-channel timing pulses with 0.3 ns resolution are generated by two Tektronix DTG5334 data timing generators synchronized by a Stanford DG535 pulse generator. The microwave pulses are amplified by an Applied Systems 127Ku TWTA; the output power at 17.5 GHz is 2 kW. The shape of the microwave pulse was monitored at the output port of TWTA via a 40 dB directional coupler. We confirmed that there was no significant distortion after amplification. The EPR signal was detected by a MRKI IQ-0618 quadrature-IF-mixer after amplification by a MITEQ AMF-5F-170180-13-10P low noise amplifier. The detected quadrature EPR signals were amplified by two IF amplifier of 300 MHz bandwidth and then acquired by an Acqiris AP240 ADC board with 1 ns resolution. The EPR resonator was a dielectric resonator (diameter: 5.59 mm, thickness: 4.60 mm, and sample access hole: 2.80 mm) made from high-purity Al_2O_3 ceramics ($\varepsilon_r = 10$) purchased from Nippon Tungsten Co., and coupled to a wave-guide by a Gordon Coupler [16] that can achieve an over-coupling condition with a loaded Q value as low as 50.

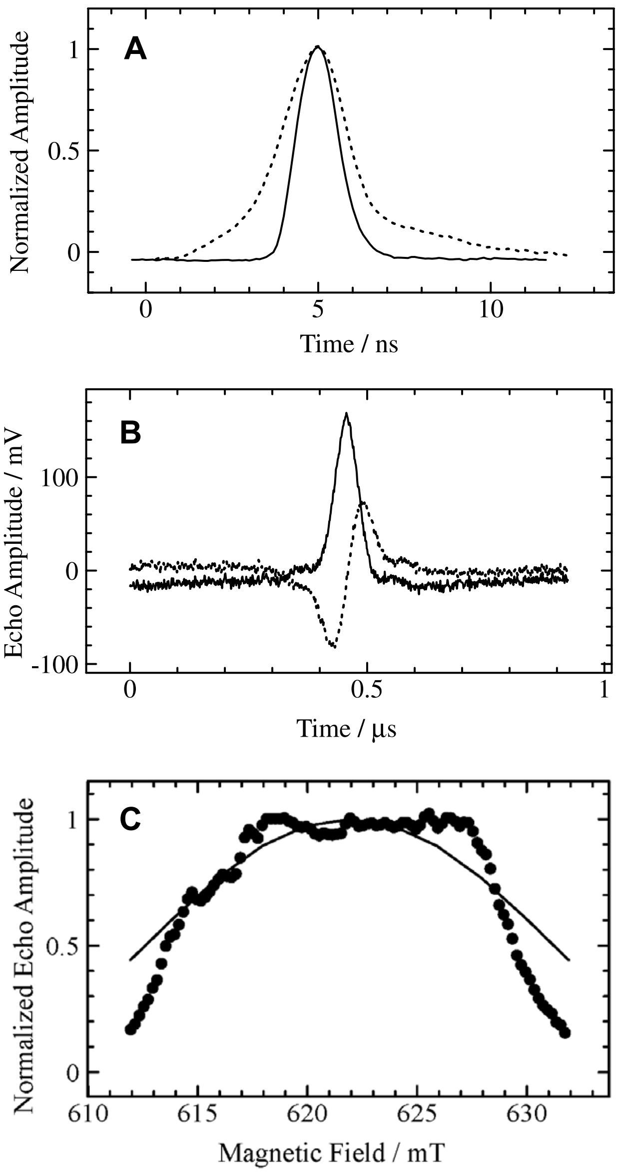

Fig. 5. Dotted and solid lines represent microwave pulses of 4.4006 GHz and 17.6025 GHz, respectively, used for spin echo measurement (A); observed in- and out-of-phase primary echo signals at room temperature represented by dotted and solid lines, respectively (B); and the closed circles and solid line are the observed and calculated echo amplitude at various magnetic field, respectively (C). The sample was γ-irradiated quartz; pulse width, 1.5 ns; microwave power, 2 kW; and pulse-to-pulse distance, 1894 ns.

shape. The removal of this small unwanted region is an improvement to the pulse shape and an additional advantage of the multiplication method.

2.4. Electron spin echo measurements

We observed electron spin echo (ESE) from the E' center of a γ-irradiated quartz sample using two microwave pulses, as shown in Fig. 5B. Since the spectral width of the E' center, 0.2 mT, is much smaller than B_1, we observed the ESE amplitude at various magnetic field at constant microwave frequency and power to obtain excitation profile, Fig. 5C. The excitation bandwidth was estimated as approximately 16 mT from the difference between the highest and lowest magnetic fields that gave 50% ESE amplitude. The microwave magnetic field B_1 was approximately 5.4 mT at saturation of TWTA (2 kW), as measured by the nutation method with a weak microwave pulse at the power 27.9 dB below the saturation level of TWTA. The turning angle of the microwave pulse of 1.5 ns is approximately 80° using this B_1 value. Assuming a simple vector model for the motion of the magnetization vector under the microwave and off-resonance Zeeman fields, [15] the excitation profile for B_1 of 5.4 mT is calculated for a Gaussian shaped EPR spectrum with the width of 0.2 mT as shown in Fig. 5C. The difference between the calculated and observed bandwidths can be attributed to the resonator Q value not being small enough. We could not observe any 'dip' using a network analyzer, but we estimated the Q value as approximately 50 from the observed bandwidth. In addition, it must be noted that the actual B_1 value is time dependent since the pulse shape is not rectangular but almost Gaussian. Therefore, the B_1 value obtained from the nutation experiment corresponds to the peak value of the Gaussian pulse.

3. Discussion

The microwave pulse width was reduced by multiplication in both the nearly rectangular and Gaussian pulses, as shown in Figs. 3B and 5A; the extent of reduction was largely consistent with that expected for the multiplication number used. To improve the technique, it is important to perform further precise investigations of the transient response of the multiplier and quantitative evaluation of the reduction effect; however, difficulties will arise in using the method based on the conventional definition of measuring the rise and fall time used in the data analysis shown in Fig. 3B.

The reduction in rise time is mainly attributed to the fact that this time corresponded to 56% amplitude, and was later than that of 10% amplitude. In contrast, the time that corresponded to 94% was later than that of 90%, and this partly canceled the reduction in rise time. Thus, the existence of opposite effects complicated the quantitative analysis. The time corresponding to 94% amplitude was highly sensitive to shape function because of the very small variation in amplitude with time. To avoid this problem, it may be preferable to use a Gaussian pulse for evaluating multiplier performance because of its simplicity, as described in Section 1.

There are two apparent disadvantages in the multiplication method. The first is the complicated nature of microwave circuitry. This is true for a homodyne system with microwave frequencies lower than the Ku band; however, for Q band (35 GHz) and higher frequencies, there are advantages in constructing a heterodyne microwave bridge based on frequency multiplication, in which case there would be no further complications resulting from the introduction of the present method. At the same time, we can achieve a wider bandwidth for higher frequencies because limitations from the Q value are also reduced.

The second disadvantage is that multiplication also increases the shot-to-shot variation in the peak amplitude, as multiplication increases the amplitude difference among pulses. To avoid this problem, input pulse power must first be well controlled, and the peak power must be close to the flat area in the high-input power region of the power dependence plot, as in Fig. 2.

Because the pulse width used in the present study is so short, we must note the frequency dependence of the group velocity of a microwave signal that propagates in a wave-guide [16]. The difference of the group velocity of the frequency components contained in a pulse signal increases the pulse width during propagation. In the present case, the pulse of 17.6 GHz carrier frequency contains approximately 17.4–17.8 GHz components. To estimate the increase in pulse width by propagating in a wave guide, we calculated the difference between the propagation times for 17.4 GHz and 17.8 GHz components. Assuming the pulse propagates 1 m length, we obtained 0.056 ns and 0.11 ns for the WR62 (15.8×7.9 mm cross section) and WR51 (13.0×6.5 mm) standard wave guides of the Ku band, respectively. Although this is not a problem in the Ku band, the difference rapidly increases with decreasing size of the wave-guide according to the increase in carrier frequency (shown in the above calculation), and can cause significant broadening of the pulse width. To avoid this problem, an oversized wave-guide should be used; this is also preferable for use with high frequencies because its loss is much smaller than that of the standard wave guide.

4. Conclusions

We successfully achieved reduction of the pulse width and observed an electron spin echo signal from the E' center of a γ-irradiated quartz sample at the Ku band frequency using a frequency multiplication method. A commercial quadruple-frequency multiplier that is not designed for pulse operation can be used, and actually reduces the rise and fall times of the microwave pulse to approximately one-half of those of the input pulse; therefore, the technique is useful in improving the limited transition times of commercial switching devices such as PIN diode switches. The technique has an additional advantage: frequency multiplication improves the pulse shape by removing the small unwanted signals that exist in the leading and following regions of the pulse signal. As is evident from the principle of operation, the present method is more advantageous at higher microwave frequencies. Frequency multiplication also has other advantages in constructing an EPR spectrometer because lower frequency devices can be used that are lower in cost and higher in performance compared to high frequency devices.

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