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High-Speed Frequency Modulation of a 460-GHz Gyrotron for Enhancement of 700-MHz DNP-NMR Spectroscopy

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Abstract The high-speed frequency modulation of a 460-GHz Gyrotron FU CW GVI (the official name in Osaka University is Gyrotron FU CW GOI) was achieved by modulation of acceleration voltage of beam electrons. The modulation speed f_m can be increased up to 10 kHz without decreasing the modulation amplitude δf of frequency. The amplitude δf was increased almost linearly with the modulation amplitude of acceleration voltage ΔV_a. At the $\Delta V_a=1$ kV, frequency spectrum width df was 50 MHz in the case of $f_m<10$ kHz. The frequency modulation was observed as both the variation of the IF frequency in the heterodyne detection system measured by a high-speed oscilloscope and the widths of frequency spectra df measured on a frequency spectrum analyzer. Both results well agree reasonably. When f_m exceeds 10 kHz, the amplitude δf is decreased gradually with increasing f_m because of the degradation of the used amplifier in response for high-speed modulation. The experiment was performed successfully for both a sinusoidal wave and triangle wave modulations. We can use the high-speed frequency modulation for increasing the enhancement factor of the dynamic nuclear polarization (DNP)-enhanced nuclear magnetic resonance (NMR) spectroscopy, which is one of effective and attractive methods for the high-frequency DNP-NMR spectroscopy, for example, at 700 MHz. Because the sensitivity of NMR is inversely proportional to the frequency, high-speed frequency modulation can compensate the decreasing the enhancement

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factor in the high-frequency DNP-NMR spectroscopy and keep the factor at high value. In addition, the high-speed frequency modulation is useful for frequency stabilization by a PID control of an acceleration voltage by feeding back of the fluctuation of frequency. The frequency stabilization in long time is also useful for application of a DNP-NMR spectroscopy to the analysis of complicated protein molecules.

Keywords Gyrotron · Second harmonic · DNP · NMR · Sub-THz · Frequency modulation

1 Introduction

Recent development of high frequency gyrotrons [1–5] using high harmonic operations and high-field superconducting magnets has achieved the breakthrough of a 1-THz operation of the gyrotrons at both short pulse modes [6, 7] and CW modes [8]. The output powers are typically 10 W to several kilowatts [9]. Such output powers are not so high as gyrotrons, because at the present, many gyrotrons for heating of nuclear fusion plasmas [10] are operating in the power range of a few megawatts in the millimeter wavelength region. However, the output power of several 10 W to several kilowatt level is still extremely high power comparing with other terahertz radiation sources of both vacuum and solid-state devices.

As the result, only gyrotrons can open the developments of the high-power terahertz technologies which are strongly required in the twenty-first century, for example, material processing [11, 12] and high-power terahertz spectroscopy [13–20]. Especially, in a recent decade, many active spectroscopy using high-power terahertz radiation sources—"Harmonic Gyrotrons" [9] were advanced and great capability of the high-frequency gyrotrons were confirmed through many applications opened only by the high-power terahertz radiation sources. For such objectives of the gyrotrons, we have begun the development of high frequency, harmonic gyrotrons, and their applications in University of Fukui in the early 1980s [9].

Among many applications of the high-frequency gyrotrons to high-power terhaertz technologies, one of most important technologies is a dynamic nuclear polarization (DNP)-enhanced nuclear magnetic resonance (NMR) spectroscopy [13–16]. As known well, NMR spectroscopy is an important and useful tool to study on the material property. However, its low sensitivity is the most severe problem. We can remove this problem by irradiation of high-power terahertz radiation at the electron spin resonance (ESR) condition and transfer of a huge magnetization of electron spins to nuclear spins. In such a way, the enhancement factor of NMR sensitivity is increased up to 1000 or more. In our experiments, we have achieved the enhancement factors of 60 for 400-MHz DNP-NMR spectroscopy [15], around 50 for 600-MHz DNP-NMR spectroscopy [14], and 30 for 700-MHz DNP-NMR spectroscopy. In the case of 600 MHz, the final enhancement factor reaches 550 by use of low temperature and low noise effects [14].

However, the factor is inversely proportional to the NMR frequency. Therefore, it is decreased, when the frequency is increased. In order to compensate this negative effect, we will try the frequency modulation of gyrotron output near ESR condition and increase the number of electron spins contributing to DNP [21]. A rough estimation results show the additional enhancement due to the frequency modulation is around factor 5.

We have already succeeded in the frequency modulation experiment using Gyrotron FU II at the rather lower frequency in sub-millimeter wavelength region [22]. In this paper, the frequency modulation results using Gyrotron FU CW GVI at the frequency of 460 GHz. This output radiation with the frequency of 460 GHz can be applied for a 700-MHz proton DNP-

NMR spectroscopy. Rough estimation results on compensation effect for decreasing of the enhancement factor at the higher frequency are also suggested briefly.

In the next section, an experimental setup and the procedure are presented. In the third sections, the experimental results on high-speed frequency modulation are demonstrated and in the final section, and a summary and future prospects are presented briefly.

2 Experimental Setup and Procedure

Figure 1 illustrates the cross section of Gyrotron FU CW GVI used for an experiment on high-speed frequency modulation, and Table 1 summarizes its designed parameters. As shown in Fig. 1, the gyrotron consists of a demountable tube, a 10-T superconducting magnet with refrigerator for cooling down the magnet to around 4.2 K and high-voltage power supply systems for electron gun. The internal mode convertor is installed in the gyrotron. Therefore, the output power can be extracted by a Gaussian-like beam. A gyrotron tube with an internal mode convertor is installed on the axis of a 10-T superconducting magnet. The power supplies for acceleration voltage of electrons and the anode voltage are Spellman's and Trek Japan Co.'s products. The superconducting magnet is a JAS TEC's 10-T magnet. In order to modulate the acceleration voltage V_a, the body of the gyrotron including a resonant cavity, a mode converter system and the output window is separated by an insulated flange (the maximum capability of the insulation is higher than 30 kV) from the collector and the electron gun and by a Teflon sheet from the superconducting magnet, so that the potential of the body can be modulated by both sinusoidal wave or triangular wave. As the result, the energy of beam electrons injected in to the cavity region is also modulated in sinusoidal or triangle waves. The modulation amplitude ΔV_a is changed up to 1 kV peak to peak. The modulation speed is increased up to 100 kHz or a little bit higher.

In Table 1, the main parameters of the gyrotron are summarized. The function of the frequency modulation is quite important, because it makes the width of ESR extended and the number of electron spins contributing to the DNP is increased. As the results, the sensitivity of an NMR spectroscopy is also enhanced. Even if the enhancement factor is decreased at higher frequency of the DNP-NMR, this effect of frequency modulation can compensate it.

After long-term operation training of the gyrotron (totally more than 100 h) and achieving the stable operation, we have tried the frequency modulation experiment. During the experiment, the vacuum in the tube is in quite good condition, the pressure is less than 6×10^{-6} Pa.

3 Experimental Results and Considerations

3.1 Measurement of the Frequency Shift as a Function of an Acceleration Voltage V_a

Before trying a high-speed frequency modulation, we have carried out the measurement on the frequency shift in a steady state as a function of an acceleration voltage V_a. In Fig. 2a, the measurement results of frequency shift are shown with changing an acceleration voltage V_a. The electron cyclotron second harmonic resonance frequency $2f_c$ is changed depending on the acceleration voltage V_a as the result of relativistic effect following the formula $2f_c=2f_{c0}/(1+V_a/511)$, where f_{c0} is the electron cyclotron frequency at $V_a=0$, V_a is acceleration voltage of beam electron presented by the unit of kilovolt. Therefore, the shift of the cyclotron frequency$\Delta f_c=f_c-f_{c0}\cong-f_{c0}V_a/511$.

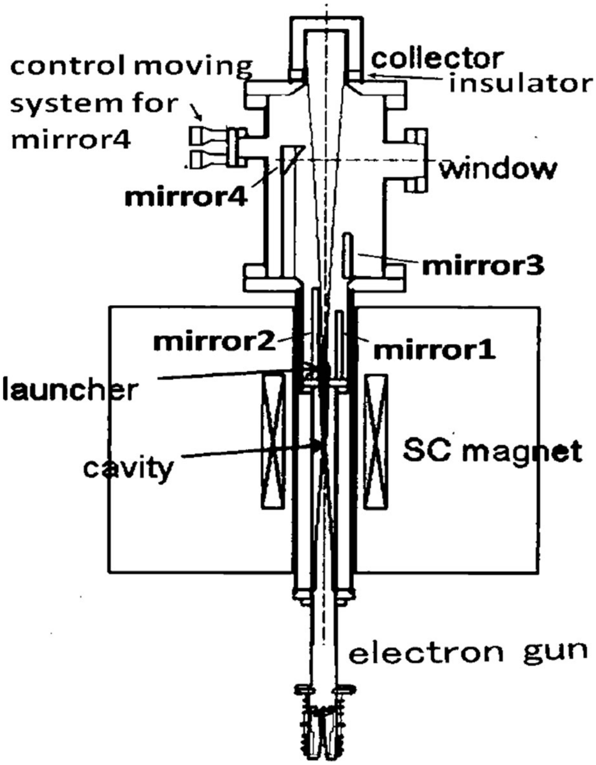


Fig. 1 The cross-section of gyrotron FU CW GVI

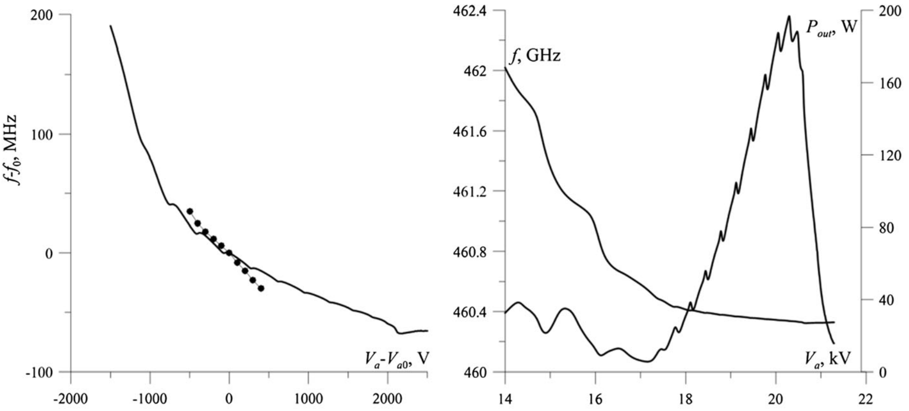
Table 1 Designed parameters of the 460 GHz gyrotron operating at the second harmonic resonance

Gyrotron tube	Demountable
Frequency f	460.4 GHz
Frequency modulation δf	100 MHz
Output power P	>30 W
Acceleration voltage V_a	20 kV
Beam current I_b	400 mA
Pitch factor α	1.2
Cavity diameter d_c	5.098 mm
Cavity length L_c	15 mm
Cavity mode	$TE_{8,5}$
Magnet type	10 T SC magnet
Field intensity at cavity B	8.55 T
Electron gun	Triode MIG
Field intensity at gun B_1	0.2 T (max)
Operation mode	CW and pulsed

However, the real frequency shift of a gyrotron output is limited and decreased by the effect of frequency pulling by the resonant cavity. A solid line in Fig. 2a shows a calculation result for the frequency shift using a self-consistent estimation system between the formulas of momentum of beam electrons and high-frequency electromagnetic waves [23]. Following parameters are used for the calculation: beam current I_b=190 mA, acceleration voltage without modulation V_{a0}=19.2 kV, the magnetic field intensity B=8.5 T, which is 0.3 % percent lower than the setting value of B considering the correction of the measurement error of magnetic field on the basis of our experimental result [24]. The calculation result is in good agreement with the experimental results; however, the slope of calculated results is smaller than that of experimental ones. Calculated output power and oscillation frequency in a wider range of beam voltage have been shown in Fig. 2b. Main four peaks correspond to four axial modes. Some small peaks located in the main peak (q=1) can be connected with the beam over-bunch. One can see that frequency modulation can be much stronger for higher axial modes. However, output power is much lower in that case.

3.2 Frequency Spectrum for Various Modulation Amplitudes of Acceleration Voltage V_a

The frequency shift of gyrotron output with variation of acceleration voltage V_a described in the previous sub-section is confirmed in steady state. If V_a will be changed in high speed of up to several kilohertz or several tens of kilohertz, it is expected that frequency high-speed modulation occurs even if the operation frequency can follow to high speed variation of V_a. We tried to apply high frequency sinusoidal modulation V_a on the body of gyrotron tube including a cavity region and mode conversion system in order to confirm if high-speed modulation of the frequency can be achieved. Figure 3 shows frequency spectra when the acceleration voltage V_a is modulated in sinusoidal mode with modulation speed f_m= 10 kHz. In Fig. 3a, the observed frequency spectrum without the modulation of acceleration voltage (peak to peak voltage ΔV_a=0) is shown. The half-value width Δf_0 is around 0.5 MHz. Figure 3b–d



a) b)

Fig. 2 **a** Frequency shift versus acceleration voltage (*points* are the experimental data, *solid* line is theory, V_{a0}= 19.2 kV, f_0=460.37 GHz); **b** calculation results of oscillation frequency and output power in wide range of acceleration voltage shows the frequency spectra under modulation of acceleration voltage with various values of ΔV_a (ΔV_a=200–1000 V). A typical shape of frequency spectrum under sinusoidal modulation is seen in this figure. The half value width for ΔV_a=1000 V is increased to $df=\Delta f-\Delta f_0\cong$ 50 MHz. Theoretically obtained spectra using quasi-static approximation are shown in Fig. 3e. The theoretical spectrum width is smaller comparing with experimental results (for ΔV_a= 1000 V, $df_{\text{theor}}\approx 38$ MHz and $df_{\text{exper}}\approx 50$ MHz) and is consistent with smaller slope for theoretical results in Fig. 2a.

3.3 High-Speed Variations of the Frequency Due to the Modulation of the Acceleration Voltage and Corresponding Frequency Spectra

Next, we tried to observe the high-speed variation of the frequency under the modulation of the acceleration voltage. In Fig. 4a, b, the frequency variation for both sinusoidal wave and triangle wave modulations of the acceleration voltage. As seen in the figures, sinusoidal wave-like and triangle wave-like modulations of the frequency are confirmed. In Fig. 5a, b, the corresponding frequency spectra are demonstrated. The formation of the spectrum is reasonable for both modulations of acceleration voltage. In addition, the widths of the frequency spectra *df* observed in Fig. 5a, b are in good agreement with the peak to peak value of the frequency variation δf observed in Fig. 4a, b. These results are confirming that the width of frequency spectrum is a complete measure of frequency variation shown in Fig. 4a, b.

The main reason of the spectrum unflatness is the dependence of power on acceleration voltage (see Fig. 2b). At this case, the voltage modulation causes also power modulation which is shown in Fig 6. One can see that the power modulation is about 20 %.

3.4 Modulation Amplitude δf as a Function of Modulation Amplitude of Acceleration Voltage ΔV_a

By changing ΔV_a, we tried to measure half-value widths of the frequency spectra as the modulation amplitude δf. The result is shown in Fig. 7 where the observed δf is plotted as a

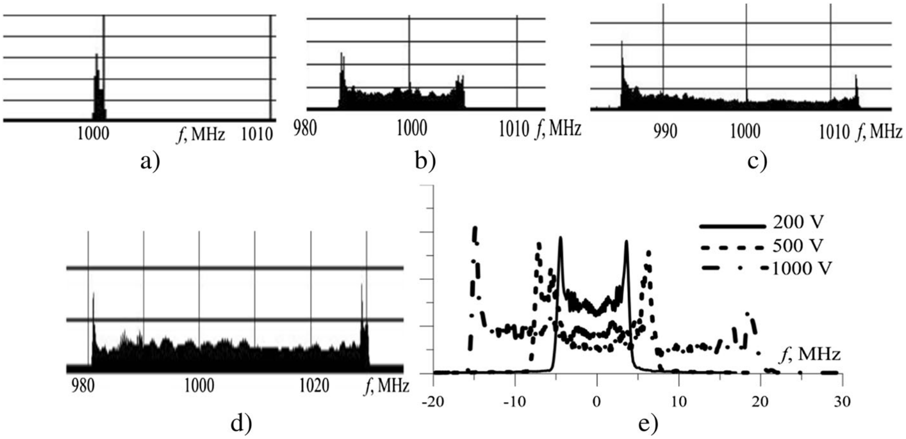


Fig. 3 Frequency spectra for various modulation amplitudes of acceleration voltage. **a** ΔV_a=0; **b** ΔV_a=200 V; **c** ΔV_a=500 V; **d** ΔV_a=1000 V; **e** calculated results with zoomed up frequency axis. V_{a0}=19.2 kV, f_0=460.37 GHz

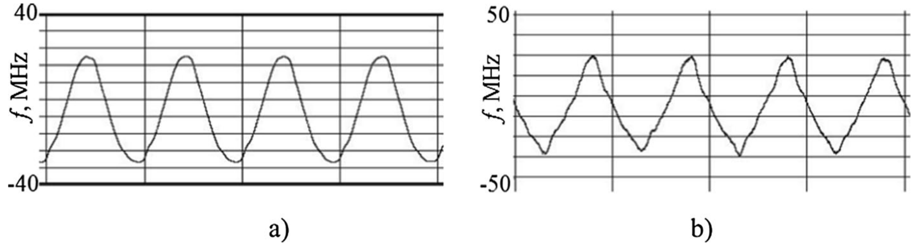


Fig. 4 Frequency versus time. fm=5 kHz, ΔVa=1 kV; **a** sinusuoidal; **b** triangle

function of the modulation amplitude of acceleration voltage ΔV_a. The modulation of the acceleration voltage ΔV_a is sinusoidal at f_m=300 Hz. It is seen in this figure the amplitude δf is almost linearly proportional to ΔV_a. The solid line is the estimation result for δf vs. ΔV_a using the self-consistent calculation formula assuming the field intensity B=8.5 T, which is 0.3 % lower than the setting value of B considering the measurement error corrected by our previous experimental result (see ref. [24]).

The estimation result is fairly good agreement with the observed result. The slope of the estimated line, $\delta f/\Delta V_a$ is around 0.05 MHz/V. This slope is much smaller than the slope of the cyclotron frequency vs. acceleration voltage, $\Delta 2f_c/\Delta V_a = -2f_{c0}/511$, which is around 0.9 MHz/V. Here, the parameters are assumed as B=8.5 T, where f_{c0}=238 GHz. This decrease of the slope of the observed value $\delta f/\Delta V_a$ results from the frequency fixed effect of the resonant cavity with high quality factor Q. The same feature is also observed in our past experiment on frequency modulation using Gyrotron FU II [22].

3.5 Frequency Modulation Under High-Speed Modulation of Acceleration Voltage V_a

We need sometime high-speed modulation of output frequency for application to high-frequency DNP-NMR spectroscopy, for example, in the case of the sensitivity enhancement experiment of the 700-MHz DNP-NMR spectroscopy. The criterion on the modulation frequency is related on the relaxation time for the transfer of magnetization from electron to nuclear. Typically, it is 10 kHz.

We tried to measure how high the modulation speed at f_m of acceleration voltage is available for effective frequency modulation. Figure 8 shows the half-value width of frequency spectrum df as a function of modulation frequency f_m with the modulation amplitude ΔV_a of acceleration voltage as a parameter. Also in this figure, there is specification of amplifier TREK 2210 that was used as supply for modulation voltage. In this figure, it is typically seen that the half-value width of the frequency spectrum ($df = \Delta f - \Delta f_0$) is increased with the modulation amplitude of

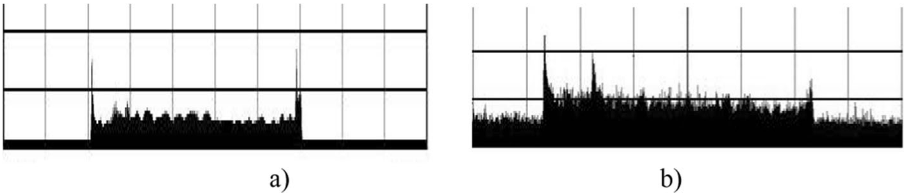


Fig. 5 Frequency spectrum. f_m=10 kHz, ΔV_a=1 kV; **a** sinusuoidal; **b** triangle, 10 MHz/div

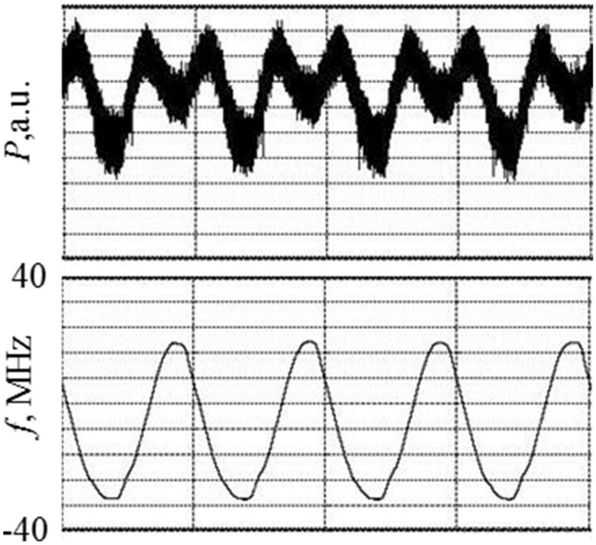


Fig. 6 Frequency modulation with sinusoidal modulation of acceleration voltage, together with the variation of amplitude. The operation frequency of gyrotron f_0=460.37 GHz. Horizontal axis 0.2 msec/div, f_m=10 kHz. Amplitude of modulated acceleration voltage 1000 V.V_{a0}=19.25 kV

acceleration voltage ΔV_a and decreased quickly with the modulation frequency f_m when f_m exceeds 30 kHz. It is consistent with the amplifier specification if we take into account that measured capacitance of gyrotron body to the ground was C=390 pF. The latter feature means that the gyrotron could not follow to so high-speed modulation f_m over 30 kHz. Also, the criterion for f_m may depend on the gyrotron characteristics, mainly the quality factor Q of the

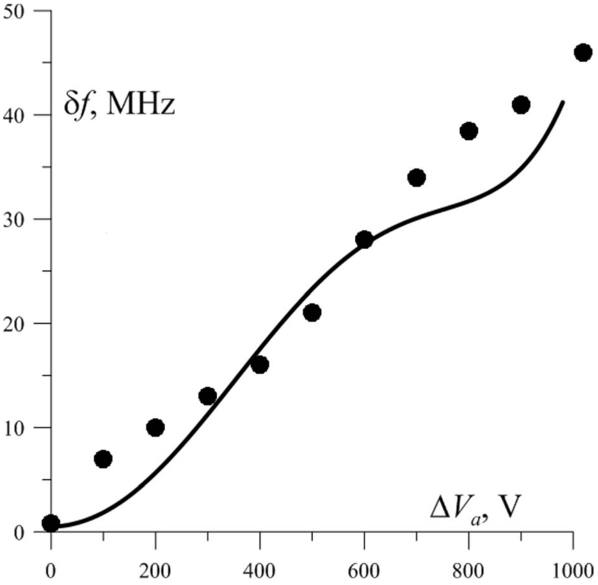


Fig. 7 Frequency modulation as a function of acceleration voltage modulation

resonant cavity. But since transients in gyrotron are typically short, the only restriction on value of f_m is amplifier. To confirm this idea, we should make the same measurements by using many gyrotrons with different Q value and compare the results each other.

We are constructing next gyrotron FU CW GIIA and including the same experiment as the above and comparing the result with the previous result.

4 Summary and Future Prospect

In order to increase the enhancement factor of the 700-MHz proton DNP-NMR spectroscopy, we tried high-speed modulation of frequency of the 460-GHz gyrotron (Gyrotron FU CW GV-I). The frequency modulation makes the ESR range wider, and as the result, the number of electron spin contributing to DNP will be increased so that the enhancement factor will be increased. The results of the experiments and the considerations are summarized as follows:

1. Before carrying out the high-speed modulation of the frequency modulation experiment, we tried measurement of the frequency shift in a steady state as a function of an acceleration voltage V_a as shown in Fig. 2a. The frequency will shift by the effect of relativistic effect on the electron mass. However, the opposite effect of fixed frequency of a resonant cavity suppressed the frequency shift. Therefore, the frequency shift rate for changing of acceleration voltage is decreased so much. Experimental result shows it is around 50 MHz/kV,

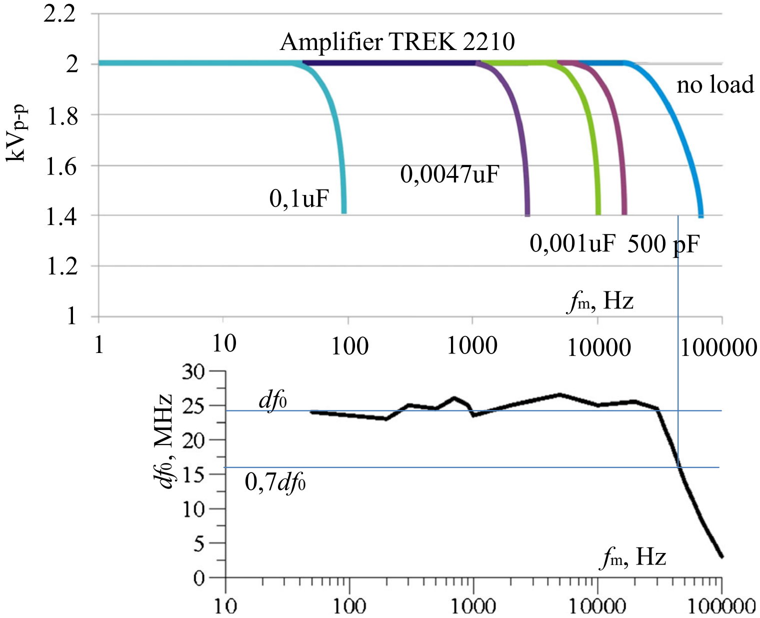


Fig. 8 The half value width of frequency spectrum df as a function of modulation frequency f_m with modulation amplitude ΔV_a of acceleration voltage as a parameter

although the simple estimation result considering the relativistic effect is around 900 MHz/kV. The calculation result using a self-consistent estimation system between the formulas of momentum of beam electrons and high-frequency electromagnetic waves is fairly good agreement with the experimental result, as shown in Fig. 2a.

2. Next, we tried to observe the high-speed variation of the frequency under the modulation of the acceleration voltage in both sinusoidal and triangle waves. The observed modulation amplitude δf is compared with the width of observed frequency spectrum under modulation. As the result, both are in good agreement. This result means that the width of the spectrum df is the complete measure of the modulation amplitude δf.
3. In next step, we tried to estimate the modulation amplitude δf of the output frequency from the measurement results of the frequency spectra. Without any modulation on the acceleration voltage, the observed frequency spectrum is a simple feature with the half-value width of 0.5 MHz. When acceleration voltage V_a is modulated sinusoidally, the feature of the frequency spectrum is changed to typical feature under sinusoidal modulation. The increase of half-value width corresponds to the increase of the modulation amplitude δf.
4. By changing the acceleration voltage δf, we studied on the dependency of the frequency modulation amplitude δf on the acceleration voltage V_a at the modulation speed f_m of 300 Hz. δf is almost linearly proportional to V_a. The rough calculation results using the same self-consistent calculation system [23] is almost in agreement with the measurement result.
5. Finally, we tried high-speed modulation of the frequency by increasing the modulation frequency f_m of acceleration voltage. The amplitude of the resulting frequency modulation is almost constant in the range of $f_m < 10$ kHz. However, f_m exceeds 20 kHz, the amplitude δf decreased gradually. This is suggesting that the gyrotron operation can follow the changing of the acceleration voltage in the case of low frequency modulation. In the case of higher-frequency modulation than criteria of f_{m0}, the gyrotron operation could not follow the changing of the acceleration voltage.

In the real experiment of high-frequency 700-MHz DNP-NMR spectroscopy [24], such a frequency high-speed modulation hopefully will contribute to increase the enhancement factor of NMR spectroscopy significantly.

The gyrotron FU CW GV-I has been already installed of 700-MHz DNP-NMR spectrometer and succeeded in the preliminary experiment and achieved the enhancement factor of around 30 without the frequency modulation. In addition, we have also succeeded in the measurements of two-dimensional 700-MHz DNP-NMR spectroscopy, which is strong tool in order to study on the analysis of the structure of complicated protein molecule in high spatial resolution.

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