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DNP-NMR spectroscopy

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Gyrotron FU CW VII for 300 MHz and 600 MHz DNP-NMR spectroscopy

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Gyrotron FU CW VII, one of the FU CW Series Gyrotrons, has been designed, constructed and completed operational tests successfully in the Research Center for Development of Far Infrared Region, University of Fukui (FIR FU). The gyrotron operates at around 200 GHz for the fundamental cyclotron resonances and at around 400 GHz for the second harmonics. These radiation frequencies will be applied to 300 MHz and 600 MHz DNP enhanced NMR spectroscopy.

1 Introduction

Our high frequency CW gyrotrons (Gyrotron FU CW Series) are high power radiation sources covering sub-THz to THz frequency region.¹⁾ The output power of such gyrotrons are much higher, by several orders than those of conventional radiation sources, for example, BWOs, TWTs, molecular gas lasers, etc. The series is now being applied for development of high power THz technologies in a wide range of fields.²⁾ A high frequency gyrotron is the only radiation source which will open the high power THz technologies such as DNP-NMR spectroscopy,²⁾⁻⁶⁾ X-ray detected magnetic resonance (XDMR) measurement,⁷⁾ ESR echo measurement in THz region,⁸⁾ etc.

Recently, short pulse gyrotrons achieved the breakthrough of 1 THz by use of high field pulse magnets.^{9),10)} The operation is in short pulse mode with the maximum pulse width being less than 1 msec.¹¹⁾ These gyrotrons exceeded the previous long-term world record of high frequency operation of gyrotron at 889 GHz¹²⁾ and reach 1 THz or higher. This success is a kind of milestone which demonstrates the possibility of gyrotrons as THz radiation sources.

However, from the viewpoint of gyrotron application to high power THz technologies, short pulse gyrotrons are insufficient in almost all cases, with CW operation often being required. In FIR FU, in order to respond to such a requirement, we are developing CW gyrotrons called Gyrotron FU CW Series covering the sub-THz to THz frequency region. We have already developed six CW gyrotrons, FU CW I to VI. Each gyrotron is designed and constructed for high power THz technology. Gyrotron FU CW VII is the seventh gyrotron included in FU CW Series and designed as a sub-THz radiation source for 300 MHz and 600 MHz DNP-NMR spectroscopy.

In this manuscript, the design and operational tests of Gyrotron FU CW VII are presented. In the next section, the profile of the gyrotron and the design principle are demonstrated, in section 3, the results of operational tests and some considerations are described and in section 4, a summary and future prospects are presented.

2. Design and construction of Gyrotron FU CW VII

For DNP-NMR spectroscopy, a high power sub-THz gyrotron is needed in order to allow transfer of the high magnetization of the electron spins (ESR) to the nuclear spins (NMR). The frequency of the gyrotron should be adjusted to the electron spin resonance frequency corresponding to the NMR frequency (or vice-versa). For 300 MHz and 600 MHz proton NMR, the corresponding ESR frequencies are 197.3 GHz and 394.9 GHz, respectively. In addition an irradiation power of several W is required. In order to respond to such a requirement, we designed Gyrotron FU CW VII operating at around 200 GHz for the fundamental electron cyclotron modes and at 400 GHz for the second harmonic mode.

In order to realize such operation frequencies, we need a superconducting magnet whose maximum field intensity is higher than 8T. We have prepared a 9.2T magnet for this purpose. The diameter of the room temperature bore is 88 mm and the uniformity of the field distribution at the center of the magnet is better than 0.1 percent within 86.4 mm in axial direction. The designed cavity modes are TE_{42} and TE_{13} modes for the fundamental operation modes and TE_{16} for the second harmonic mode.

A schematic cross-section and the side-view of the gyrotron are shown in Fig. 1 and Photo 1, respectively. The diameter and the length of the cavity are 4.35 mm and 19 mm, respectively. The frequencies of designed modes are 186.97 GHz (TE_{13} mode), 203.75 GHz (TE_{42} mode) and 395.28 GHz (TE_{16} mode). Typical parameters of electron beam are as follows,

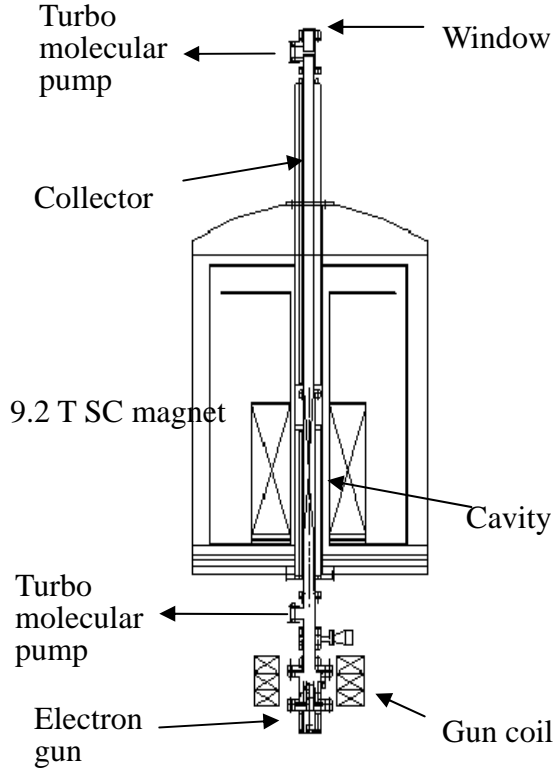


Fig.1 A cross-section of Gyrotron
FU CW VII



Photo 1 Side-view of Gyrotron FU CW VII

acceleration voltage $V_b=10-15$ kV, beam current $I_b=200 - 350$ mA, duty ratio $\eta=0.1 - 1$ (CW) and the repetition rate $f_m=1-10$ Hz.

Fig. 2 shows the starting currents I_{st} as a functions of magnetic field intensity B for many cavity modes operating at both the fundamental and the second harmonics. The designed cavity modes TE_{42} , TH_{13} and TE_{16} can be excited when the beam current is higher than the starting current I_{st} . A beam current of 200 mA is enough for excitation of all the designed modes when the magnetic field intensity B is adjusted to the optimum condition.

3. Operation test results and consideration

After constructing the gyrotron, we have tested the operation using high voltage power supplies for a triode magnetron-injection electron gun.

3-1 Measurement of radiation power as functions of magnetic field intensity B

Fig. 3 shows the radiation power for the fundamental operations measured at the open end of circular waveguide system by a pyro-electric detector and the corresponding starting current

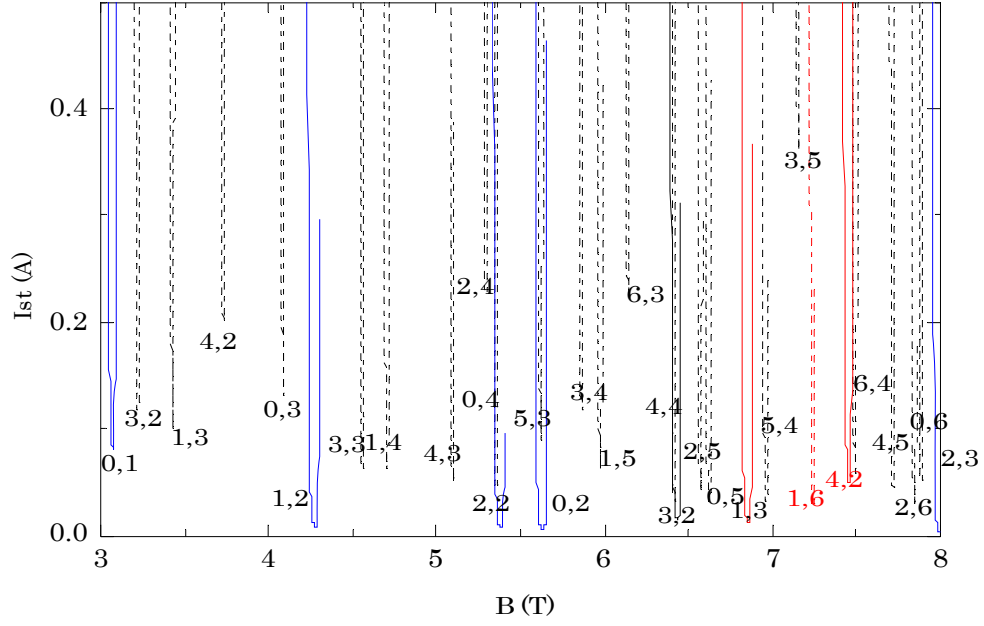


Fig. 2 Starting current I_{st} as functions of magnetic field intensity. Solid lines show the fundamental operation and broken lines the second harmonic operation. The acceleration voltage $V_k = 12$ kV.

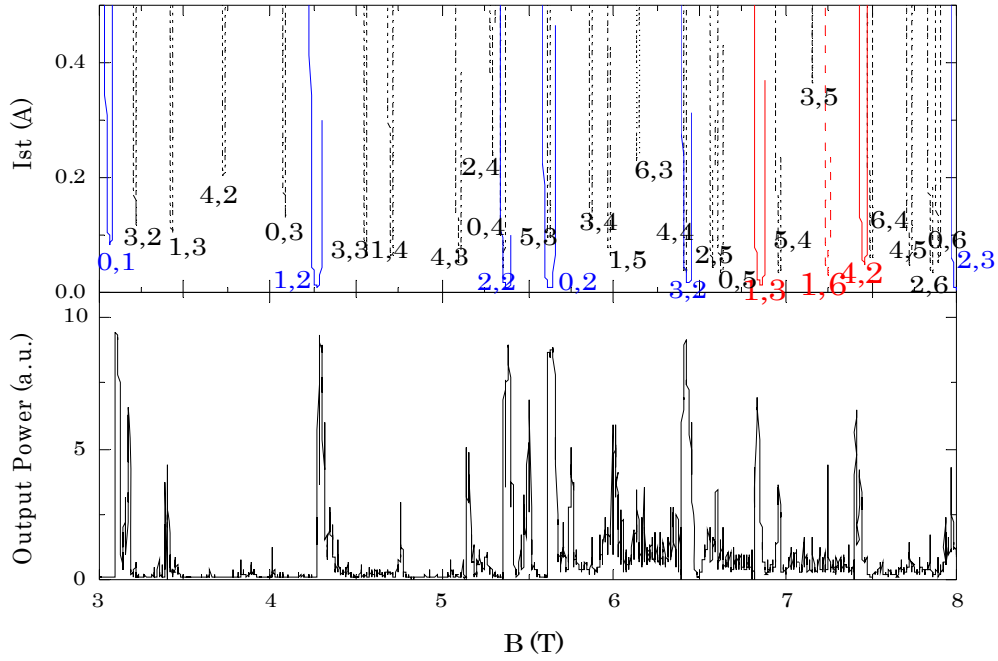


Fig. 3 Radiation power for the fundamental operation measured at the open end by a pyro-electric detector and the corresponding starting current for each cavity mode.

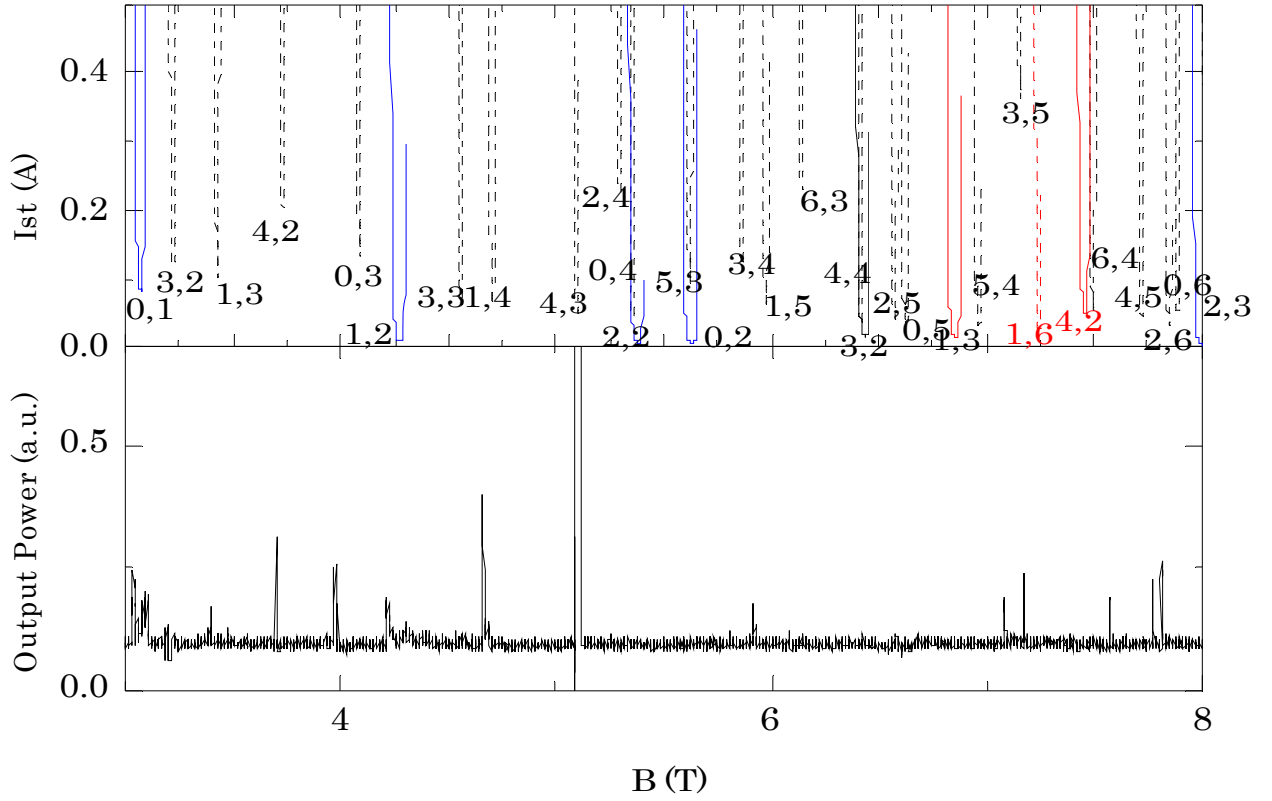


Fig. 4 Radiation power for second harmonic operation measured at the open end by a pyro-electric detector and the corresponding starting current for each cavity mode.

for each cavity mode. Fig. 4 shows the radiation power of the second harmonic operations and the corresponding starting current. Many radiation peaks appear, as the magnetic field is varied. The upper trace is the direct measurement while the lower trace shows the power after a high pass filter, which has narrow circular hole of diameter 0.7 mm with corresponding cut-off frequency of 251GHz. In the magnetic field range from 6 T to 8 T, radiation from the fundamental operations is removed and as a result, only radiation from the second harmonic is observed.

Radiation peaks coming from the designed cavity modes TE_{13} and TE_{42} are observed only in the upper trace at the field intensities of 6.83 T and 7.42 T. While, the radiation peak from the cavity mode TE_{16} appears in both traces at the field intensity of 7.22 T.

Tables 1 and 2 summarize measurement results for all radiation peaks appearing in both traces, that is, the direct measurement and the measurement after high-pass filters. Here, we used two filters whose diameters are 1.1 mm and 0.7 mm and applied each of them to the

Table 1 Parameters of the fundamental operation modes.

mode	B_{cal} (T)	B_{meas} (T)	f_{cal} (GHz)	f_{meas} (GHz)	R (m)	f_{est} (GHz)	P (W)
TE ₀₁	3.07	3.04	86.28			86.11	
TE ₁₂	4.25	4.23	117.15			116.92	
TE ₂₂	5.35	5.32	147.28			146.99	
TE ₀₂	5.62	5.55	154.06			153.76	
TE ₃₂	6.46	6.35	175.97			175.63	
TE ₁₃	6.85	6.83	187.4	187.08	4		213
TE ₄₂	7.45	7.42	203.75	203.30	5		138
TE ₂₃	8.00	7.89	218.82			218.39	
TE ₀₃	8.17	8.06	223.29			222.85	

Table 2 Parameters of the second harmonic operation modes

mode	B_{cal} (T)	B_{meas} (T)	f_{cal} (GHz)	f_{est} (GHz)	P (W)
TE ₀₆	7.89	7.82	430.38	429.54	
TE ₂₆	7.84	7.78	428.12	427.29	
TE ₄₅	7.72	7.57	421.17	420.35	
TE ₁₆	7.25	7.22	395.28	394.51	27
TE ₃₆	7.15	7.06	391.96	391.19	
TE ₁₅	5.97	5.91	326.15	325.51	
TE ₃₄	5.86	5.82	320.72	320.09	
TE ₄₃	5.09	5.00	278.30	277.75	
TE ₁₄	4.70	4.66	258.90	258.39	
TE ₀₃	4.09	3.97	223.74	223.30	
TE ₄₂	3.73	3.70	204.73	204.33	
TE ₁₃	3.43	3.39	187.40	187.03	

corresponding magnetic field range where it operates as a high pass filter.

In both tables, the cavity modes are shown together with, the calculated resonant frequencies of the cavity modes f_{cal} , calculated field intensities B_{cal} for the cavity modes, measured field intensities B_{meas} where the cavity modes are excited. The frequency was measured by a heterodyne detection system only for designed cavity modes TE₁₃ and TE₄₂. The measured frequencies f_{meas} are a slightly lower than the calculated frequencies f_{cal} . The difference may come from a fabrication error in the cavity radius R . The error ΔR estimated from the

frequency differences are $\pm 4.5 \mu\text{m}$. In both tables, the frequencies f_{est} estimated by taking this error into account are presented. We assume that real radiation peaks occur at these estimated frequencies f_{est} . In addition, the output power P measured by a water-load for the three designed cavity modes are given. For fundamental operation, P is several hundred W, while, for second harmonic modes, P is several tens W. Both of these powers are enough for application to DNP-NMR spectroscopy, because the irradiation power needed on the sample is only a few W or less.

3-2 Measurement of output power for designed cavity modes

For application of Gyrotron FU CW VII to DNP-NMR spectroscopy, we need to control the output power and adjust it to the optimum value, because too high power will heat up the sample and too low power will not induce sufficient transfer of magnetization from the electron spins to the nuclear spins. We have therefore measured the output power of the designed cavity modes as a function of beam current.

Fig. 5, shows the dependency of the output power on the beam current at the optimum magnetic field with a constant acceleration voltage of 15 kV. For the fundamental operation of designed cavity modes TE₄₂ and TE₁₃, the output power increase with beam current I_b and saturate at around $I_b = 250 \text{ mA}$. The saturation power levels are 130 W and 150 W respectively.

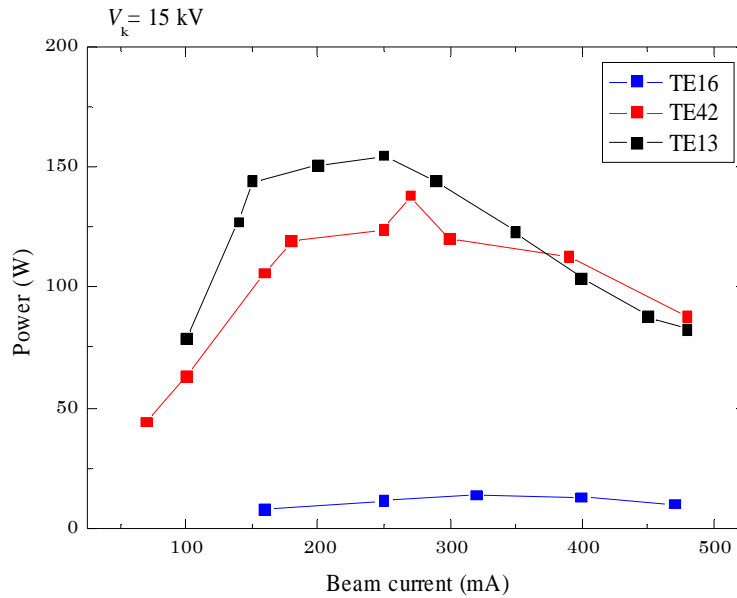


Fig. 5 The measured output power for the three designed cavity modes as a function of the beam current

In the case of the second harmonic operation of the cavity mode of TE₁₆, the output power is increased with beam current and saturated at a slightly higher beam current at around 30 W. In any case, we can control the output power by controlling the beam current and can adjust it to the optimum value for DNP-NMR measurement.

3-3 Measurement on the frequency spectra of radiation from the designed cavity modes

The quality of the gyrotron operation is also important for its application to DNP-NMR spectroscopy. We have checked the frequency spectra of the output powers from both fundamental operations of TE₁₃ and TE₄₂ cavity modes. Photo 2 shows the frequency spectra observed by a heterodyne detection system which consists of a synthesizer, a harmonic mixer and a spectrum analyzer. Photo 2 (right) and (left) demonstrate frequency spectra of radiations from TE₁₃ and TE₄₂ cavity modes. In both spectra, the half value width is several MHz. The center frequency looks stable with fluctuation of the frequency being less than 1 MHz.

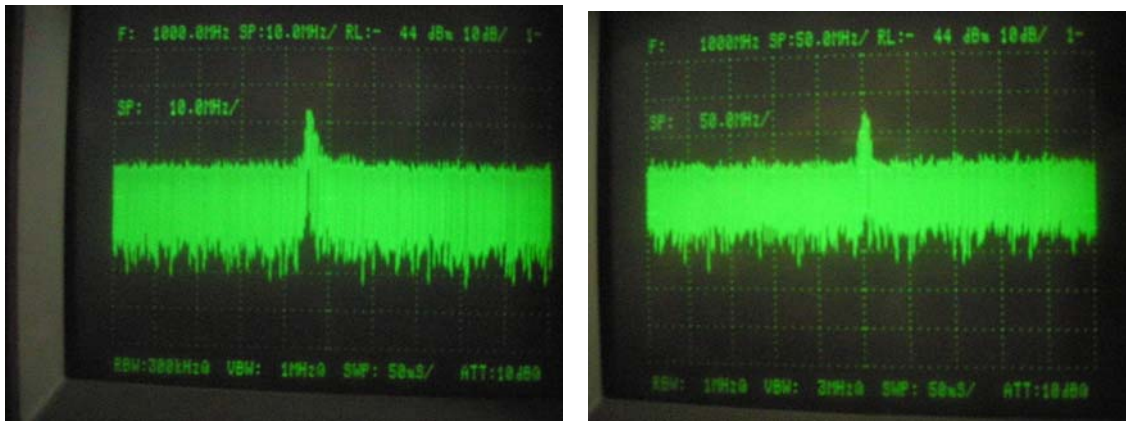


Photo 2 Frequency spectra of the designed modes TE₁₃ (right) and TE₄₂ (left) operating at fundamental resonance. One division of horizontal axis is 10 MHz (right) and 50 MHz (left), respectively.

This means the frequency stabilization $\delta f/f$ is better than 5×10^{-6} which is important for DNP-NMR spectroscopy.

3-4 Mode purity of gyrotron output

Usually, in a gyrotron, the power emitted from the cavity transmits in an oversized waveguide until it is launched from the output window. Although the mode is pure just after the cavity output, it is disturbed by mode conversion during transmission in an oversized waveguide so it is expected that mode purity becomes worse at the output window.

We have measured the mode pattern of the radiation launched from the window. From the observed pattern we can obtain some information about mode purity at the output window. In Fig. 6, the observed radiation patterns above the window for the three designed cavity modes are shown. A sheet of vinyl chloride was placed in the radiation beam and the temperature increase on the sheet was observed by an infrared camera. The temperature increase is approximately proportional to the injected radiation power, so we can observe the distribution of radiation power on the sheet. In this way we can obtain the radiation patterns shown in Fig.6.

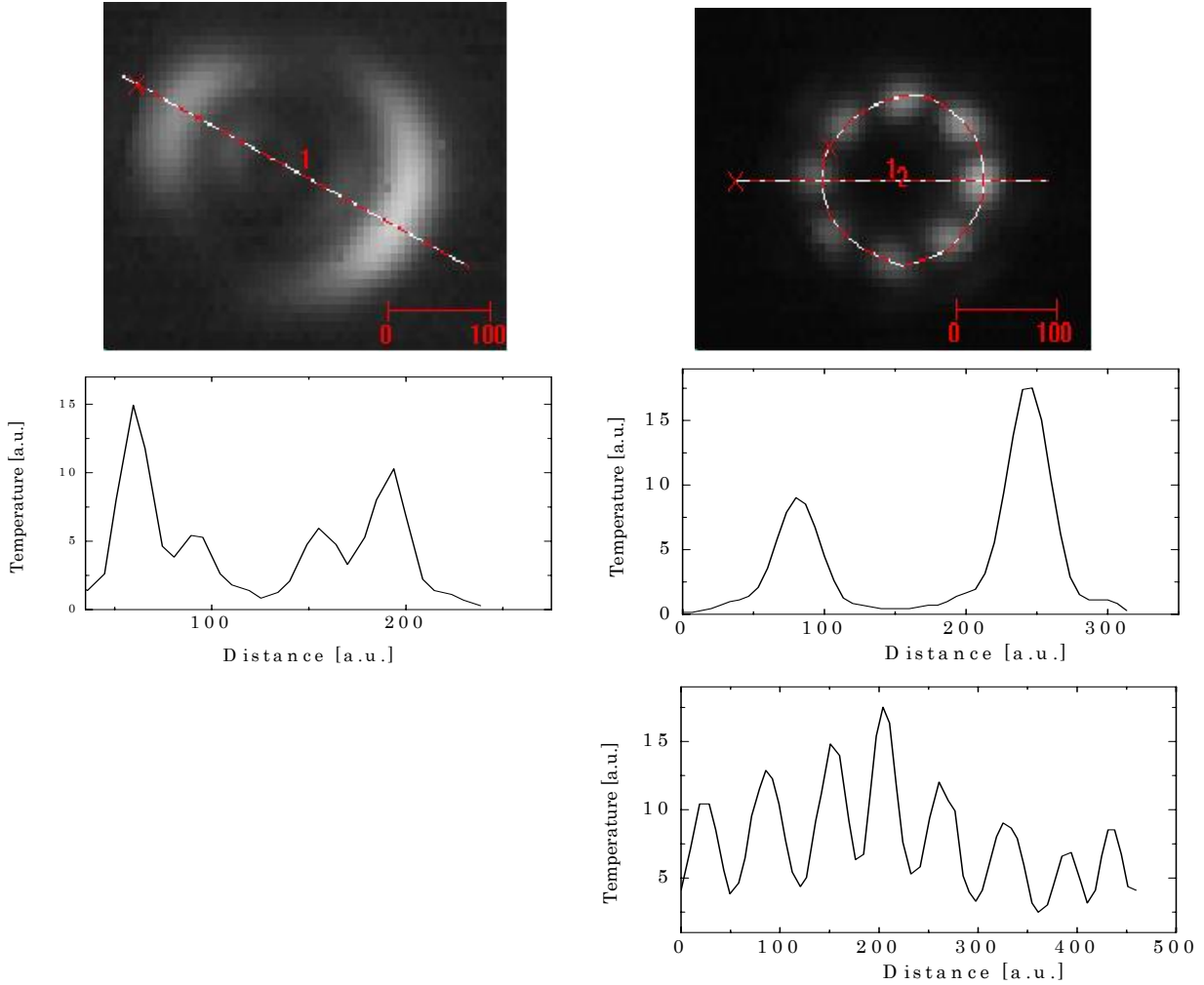


Fig. 6 (a) Radiation patterns for TE₁₃ mode (left) and TE₄₂ mode (right) of the fundamental operations. Lower traces show temperature distributions measured on a sheet of vinyl chloride along the broken lines shown in the upper patterns, which are a measure of the radiation power distribution.

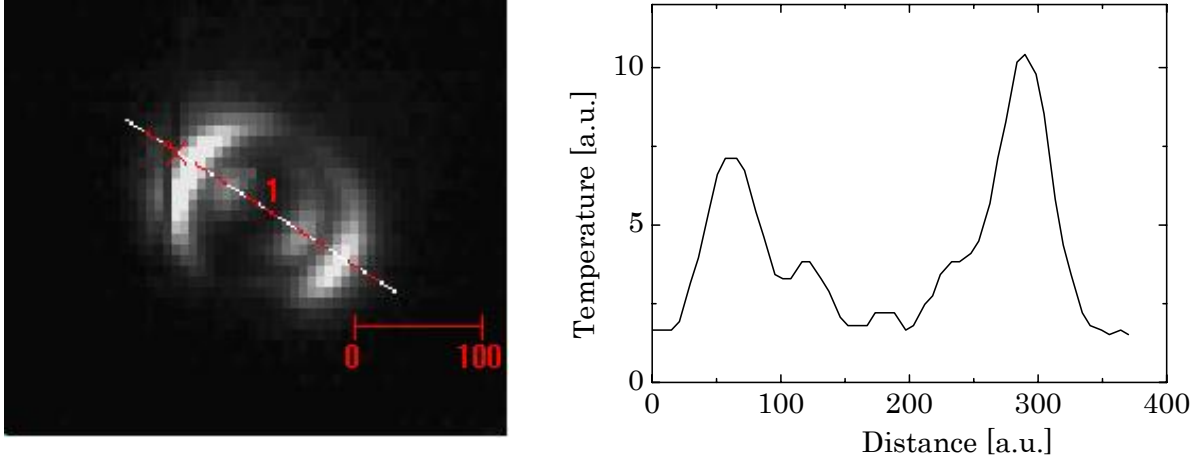


Fig. 6 (b) Radiation pattern for TE_{16} mode at the second harmonic operation. The trace on the right hand side shows the temperature distribution measured on a sheet of vinyl chloride, which are a measure of the radiation power distribution.

As well as the radiation patterns, the power distribution along the broken lines is shown. In the case of TE_{13} mode, five (2 large and 3 small) peaks appear in the radial direction. This means that the main mode included in the pattern is TE_{13} . In the case of TE_{42} mode, eight peaks appear in the azimuthal direction and two peaks in the radial direction. This means that the main mode is TE_{41} and that mode conversion occurs from TE_{42} to TE_{41} . In the case of TE_{16} mode, five peaks appear in the radial direction. This means that mode conversion occurs from TE_{16} to TE_{13} .

We are intending to convert the gyrotron output to a Gaussian beam in the future. In that case, high mode purity is required in order to obtain a high quality Gaussian beam. We will try to carry out the conversion for the present gyrotron output.

3-5 Stability of the output power

Stability of output power is one of most important requirements for the gyrotron as a radiation source of DNP-NMR spectroscopy. Fig. 7 shows the variation of the output power as a function of time. In the first 40 minutes, the output power P varies drastically, because of variation of beam current I_b as the line voltage varies. In the next 80 minutes, the line voltage becomes much more stable, and as the result, the variation of the output power P is smaller. During these 80 minutes the variation $\Delta P/P$ is less than 5 percent ($\Delta P/P < \pm 0.05$). These results mean that the output power can be stabilized by stabilization of the line voltage even in the free running of the gyrotron.

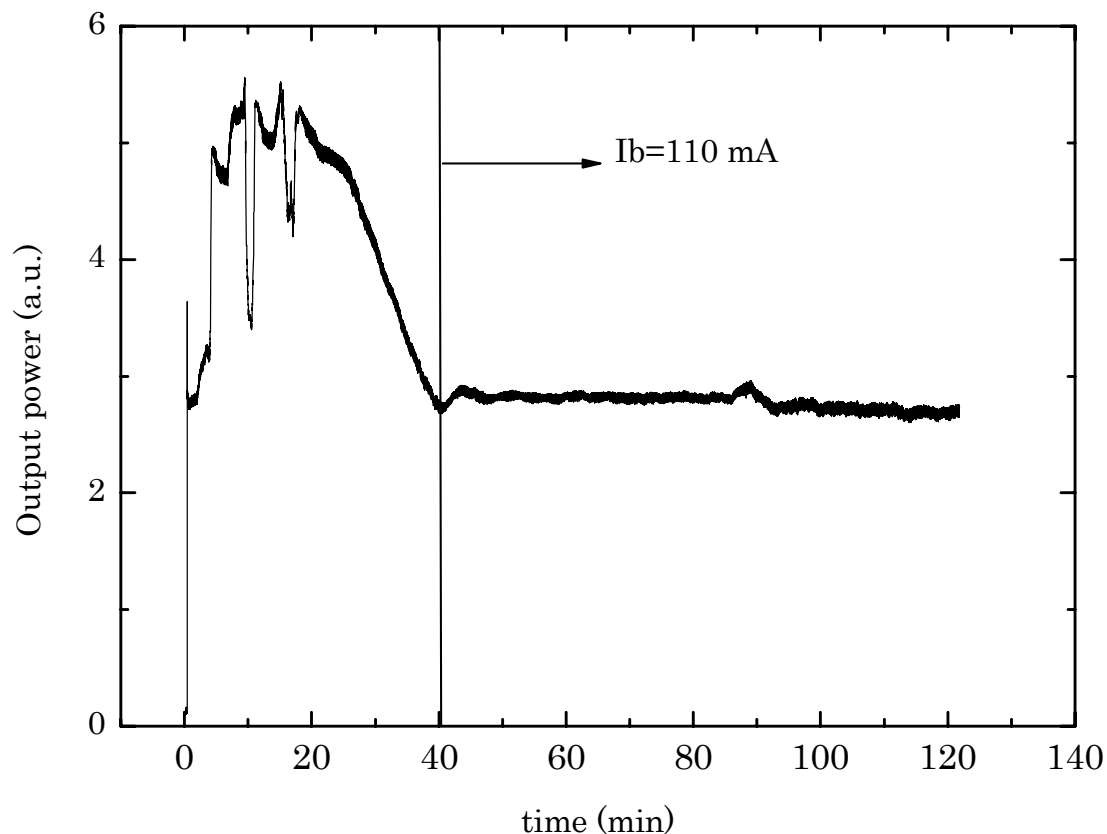


Fig. 7 Variation of output power for TE_{13} mode with the time. In the first 40 minutes, the power varies drastically because of variation of the line voltage. However, in the next 80 minutes, the line voltage is stabilized much improving the stability. The stability is better than 5 percent.

We have already installed Gyrotron FU CW VII on the 300 MHz and 600 MHz NMR spectrometer in the NMR group, University of Warwick, UK. (See Photo 3) The output frequencies of the designed cavity modes are slightly different from the frequencies required for 300 MHz and 600 MHz DNP-NMR spectroscopy. However, the frequency of NMR can be adjusted to the gyrotron frequencies by changing the NMR field and frequency.

The output powers achieved for the three designed cavity modes are enough for the purpose. In addition, the stability of frequency and output power can be achieved by controlling the beam parameters.

Gyrotron FU CW VII should allow the high enhancement of NMR sensitivity by Dynamic Nuclear Polarization (DNP).



Photo 3 Gyrotron FU CW VII has already been installed on a 300 MHz and 600 MHz NMR spectrometer at University of Warwick.

4 Summary and the future prospects

Gyrotron FU CW VII is one of sub-THz CW gyrotrons included in Gyrotron FU CW Series developed in FIR FU. It was designed for study of DNP enhanced NMR spectroscopy by the NMR Research Group, University of Warwick, UK in collaboration with FIR FU. The NMR spectrometer is tunable to both ~ 300 MHz and 600 MHz proton frequency and Gyrotron FU CW VII was designed accordingly. The designed cavity modes are TE_{13} and TE_{42} for fundamental operation and TE_{16} mode for second harmonic operation. Resonant frequencies of these modes are 186.97GHz, 203.75GHz and 395.28 GHz, respectively. The first frequency will be applied to ~ 287 MHz proton DNP-NMR measurement, while the third frequency will be used for 600 MHz DNP-NMR measurements.

The diameter and the length of the cavity are 4.35 mm and 19 mm, respectively.

The gyrotron was constructed and tested by using a 9.2 T superconducting magnet.

- 1) The frequency is step-tunable over a wide range because there are many cavity modes at both fundamental and second harmonic cyclotron resonances. The frequency range is from 86 GHz to 223 GHz for fundamental operation and from 187 GHz to 430 GHz for second harmonic operation.
- 2) The observed frequencies of the designed modes TE_{13} and TE_{42} are 187.08 GHz and

203.30 GHz, respectively. These frequencies are slightly lower than the designed frequencies. The difference may come from the fabrication error ΔR of the radius of the cavity. Comparing the observed frequencies with the designed frequencies, the error ΔR can be estimated as $\Delta R \sim 4.5 \mu\text{m}$. The frequency of the third designed mode TE_{16} could not be measured, because our heterodyne system is not available for the frequency. However, we could estimate it by taking the fabrication error ΔR in account. The estimated frequency is 394.51 GHz.

- 3) Results of output power measurement show that in both fundamental and second harmonic operation the output power can be controlled by controlling the beam current. The saturation levels of the power are higher than 130 W for fundamental operation and around 30 W for second harmonic operation. These powers are enough for application to DNP-NMR spectroscopy.
- 4) Measurement of the frequency spectra shows that the half value widths are a few MHz and the fluctuation of frequency is less than 1 MHz. This means the short term stability of the frequency is higher than 5×10^{-6} .
- 5) In order to study the mode purity at the output window, we have observed the emission pattern above the window. In some cases, mode conversion to a lower radial mode occurs. However, the three designed modes could be converted to the Gaussian modes by use of a specially designed quasi-optical conversion system.
- 6) Stability of the output power for a longer time has been checked in the free-running of gyrotron operation. The result shows that fluctuation of the line voltage causes the fluctuation of output power because of the fluctuation of beam current. However, when the line voltage was stabilized, the output power was also stabilized. The stability is higher than ± 5 percent during 80 minutes operation.

Gyrotron FU CW VII is now installed on the NMR device at University of Warwick, UK. The DNP-NMR measurement at 300 MHz and 600 MHz will start soon. For future applications we need a frequency continuously tunable gyrotron for DNP-NMR spectroscopy so the frequency can be adjusted to the optimum value. Such gyrotrons are now being developed in MIT⁵⁾ and FIR FU.¹³⁾

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