

The Microwave Sources for EPR Spectroscopy

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Abstract—Rapid development of many scientific and technical disciplines, especially in material science and material engineering increases a demand for quick, accurate and cheap techniques of materials investigations. The EPR spectroscopy meets these requirements and it is used in many fields of science including biology, chemistry and physics. For proper work, the EPR spectrometer needs a microwave source, which are reviewed in this paper. Vacuum tubes as well as semiconductor generators are presented such as magnetron, klystron, traveling wave tube, backward wave oscillator, orotron, gyrotron, Gunn and IMPATT diodes. In this paper main advantages of gyrotron usage, such as stability and an increased spectral resolution in application to EPR spectroscopy is discussed. The most promising and reliable microwave source is suggested.

Keywords—electron paramagnetic resonance, gyrotron EPR, microwave sources, orotron, terahertz waves.

1. Introduction

Electron paramagnetic resonance (EPR) is a very sensitive and specialized method that can be applied for both element and chemical reaction investigations. The EPR method can be used to detect the organic and inorganic compounds in electrochemical systems. A form of the equation, describing an EPR resonance condition:

$$h\nu = g\beta B, \quad (1)$$

shows that an EPR signal can be observed in two ways.

The first is an observation of the resonant energy absorption versus frequency ν of the electromagnetic field at a constant magnetic induction B . The second way is to observe the resonant energy absorption versus magnetic induction B of the constant magnetic field at a constant frequency ν (g is Lande factor).

Experiments using EPR are mainly conducted in X and Q bands, and less frequently in V and W ones. The common use of the X and Q bands is due to their wide availability and the relatively low price of all microwave components (developed for radar systems). The second reason for the wide use of the X and Q bands is the use of comparatively weak constant magnetic fields with an induction of the order of 1 T, which is achievable with conventional electromagnets. Development of such electromagnets is relatively

simple and cheap. Their operation is much more convenient than superconductive electromagnets, for instance. However, a spectroscopic splitting factor for these bands limits the ability to examine the substances, which exhibit low magnetic anisotropy.

Measurements carried out in the millimeter wave band in a frequency range above 40 GHz introduce a high resolution of a spectrum for a given g coefficient. Spectral resolution increases with radiation frequency and the intensity of the constant, resonant magnetic B_0 . It has been demonstrated in past work [1] based on analysis of different biological, polymeric and other spin systems examined in the D band. There may be several criteria for selecting a millimeter wave source for EPR, including performance, output power, tuning bandwidth, durability, convenience and costs [2]. Solid-state sources such as Gunn and IMPATT diodes are often chosen because of their relatively low cost and compact dimensions. Due to them having small dimensions, it is easier to integrate them into EPR devices. What is more, solid-state sources do not require high control voltages and additional cooling. However, they can only deliver low output power compared to vacuum tubes.

Table 1
Example bands used in EPR spectroscopy

Band	L	X	K	Q	V	W	D
λ [mm]	300	30	12.5	8.5	5.6	3.2	2.1
f [GHz]	1	10	24	35	65	95	140
B_0 [T]	0.04	0.36	0.86	1.25	2.3	3.39	5.00

2. Sources of the Microwave Radiation

2.1. Magnetron

Magnetrons were the first microwave devices used for the generation of high frequency radiation. The first magnetron was constructed by Albert Hull in 1920. A modern project of the magnetron device was proposed during the Second World War in 1940 by John and Harry Boot from the University of Birmingham. Engineers managed to construct radars, which despite their pulsed operation contributed to the victory of the Allies over Germany. Nowadays, most magnetrons are used in households, where they work as

a source of the microwaves in microwave ovens, whereas in radar applications the most frequently used sources are klystrons and backward-wave tubes.

The magnetron can generate high power radiation [3] (up to hundreds of kilowatts) and high frequency, which is limited mainly by the intensity of a constant magnetic field according to the formula

$$f = \frac{eB}{mc}, \quad (2)$$

where f denotes the frequency of the electron oscillations, e is the charge of the electron, B is the induction of an external constant magnetic field, m is the mass of the electron and c is the speed of light. The magnetron is characterized by a high instability of both a generated frequency and its phase [4]. Due to these disadvantages, magnetrons are not used in EPR spectroscopy.

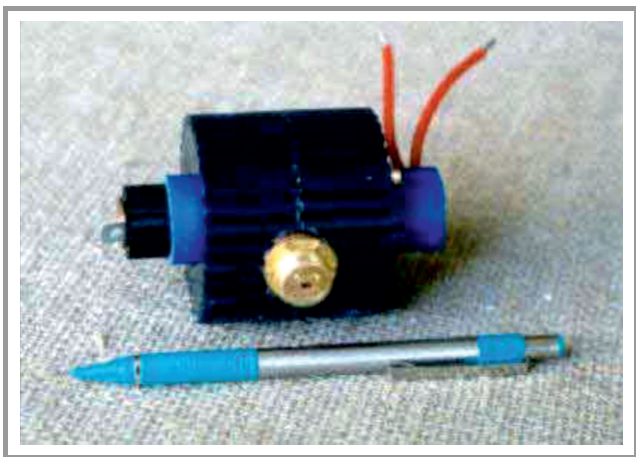


Fig. 1. The SHM magnetron delivers 1 kW output power at 95 GHz [8], [10].

Nevertheless, the constant progress in magnetron development for higher frequencies and output powers can be noticed. In the most high frequency magnetrons a new type of this device is used, the so-called spatial-harmonic magnetron (SHM), which utilizes a cold secondary-emission cathode. It was first proposed by Robertshaw [5] and lately reintroduced by [6], along with the whole theory for multimode interactions, formation of doublets, and transient processes. Based on this theory several devices were designed and built for 95 GHz (Fig. 1) [7] and 210 GHz [8]. Another magnetron for higher frequency (320 GHz) is under development [9]. The SHM magnetrons could open a new age of magnetrons and despite their disadvantages they have very important advantages such as their small size and low price in comparison with other microwave tubes, e.g. klystrons and gyrotrons.

2.2. Gunn Diode

Historically, one of the first semiconductor sources of microwave radiation was a Gunn diode [11]. It was used as

an active element in many microwave generators and amplifiers. Thanks to its simple construction and an ability to generate oscillations at very high frequencies, it is used in many systems. Diodes based on gallium arsenide are able to generate radiation at a frequency reaching 200 GHz, while frequency reachable by the diodes based on gallium nitride can be as high as 3 THz [12]. Moreover, Gunn generators are tunable in a wide frequency range [13]. Unfortunately, the achieved power is low and does not exceed several milliwatts CW. However, this power level is sufficient to be used in EPR spectroscopy.

As was written in [2], Gunn diodes can provide up to 100 mW CW radiation at 95 GHz and 20 mW at 140 GHz. The main disadvantage of solid-state sources is poor noise and frequency jitter characteristics, which must usually be compensated by phase-lock loops to a reference oscillation source or external cavity. For example, the Gunn diode working at 95 GHz has a phase noise of about -40 dBc/Hz at a 10 kHz frequency offset. However, phase locking limits the sweep ability of the necessary microwave sources e.g. for control matching the EPR cavity.

The diode based oscillators can be expected to work continuously for up to 100,000 h, which is a much longer working time than vacuum tubes could offer.

2.3. Klystron

Klystrons, as well as magnetrons, were invented at the beginning of the XX century. The main work was done by the Varian brothers [14]. Klystrons can work as amplifiers as well as microwave generators for a wide range of wavelengths (from radio frequencies up to upper microwaves). Klystrons are widely used in such areas as radars, TV transmitters, satellite communication and in modern particle accelerators [15].

A principle of klystron operation is based on a conversion of the energy of accelerated electrons to the energy of the electromagnetic field. This conversion occurs due to the bunching of electrons in the alternating electric field. A klystron usually consists of an electron gun, a resonance cavity (or several cavities) and a collector or reflector. In a typical generation klystron, which is the reflex klystron [16].

Klystrons are stable sources of electromagnetic radiation, which can be successfully used in EPR spectroscopy [17]. In a typical EPR instrument, a klystron tube is used to generate monochromatic microwave radiation in the X band (≈ 9.75 GHz), but all other bands are also used.

Although they also have limits. The main limit for klystrons is the highest frequency, which can be generated at a reasonable power level (tens of watts). The ratio between the generated frequency and the output power for modern klystrons drops rapidly when working frequency reaches 100 GHz and above [18] (Fig. 2).

One solution to the problem of output power loss generated by klystrons was the development of the so-called

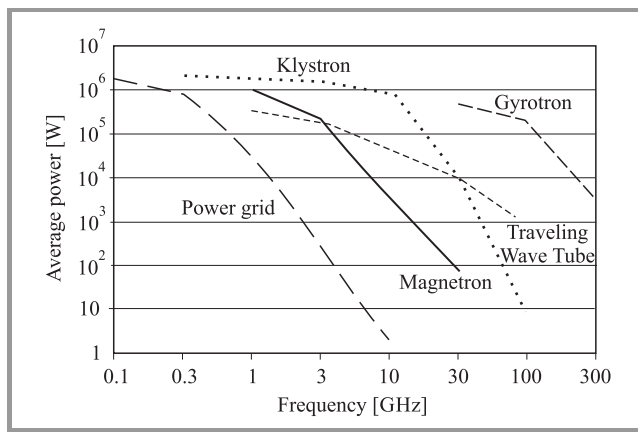


Fig. 2. Diagram presenting power versus frequency limits for microwave generators [18].

extended interaction klystrons (EIK), which combine the advantages of normal klystrons and those of traveling wave tubes (TWT) [19]. It is said that EIK klystrons will reach very high frequencies (up to 1 THz), and at the same time with a high power level and frequency stability. In the present day, there is research on klystrons that will work on a frequency of 670 GHz [20].

2.4. Traveling Wave Tube

As with the previously mentioned radiation sources, the travelling wave tube (TWT) was also invented during the Second World War [21]. It is said that the creators of these kinds of tubes are Rudolf Kompfner and Nils Lindenblad. The principle of operation is similar to klystrons, which is also based on extraction of energy from accelerated electrons and the transfer of part of it to the electromagnetic field. However, in this case the delaying structure is used, which causes electrons to move with a velocity close to the alternation of the electromagnetic field. Electrons are periodically focused by magnets or electromagnets, which, as mentioned before, cause the occurrence of the bunching of electrons.

TWTs are usually used as amplifiers of microwave radiation and they are characterized by a wide bandwidth, which make TWTs very good broadband amplifiers. In contrast to klystrons, TWTs are able to reach much higher output powers at frequencies above 100 GHz. Due to this fact, TWTs are broadly used in many areas such as, among others, telecommunications, radar techniques and EPR [22].

2.5. Backward Wave Oscillator (BWO)

A backward wave oscillator (BWO), also called a backward wave tube, is a vacuum tube that was invented by the inventor of the traveling wave tube, Rudolf Kompfner [21]. He demonstrated O-type BWO in 1951, whereas M-type was presented by Bernard Epsztein [23] in the same year. The BWO is used to generate microwaves up to the terahertz

range. It also belongs to the traveling-wave tube (TWT) family and is an oscillator, which can be tuned in a wide electronic range.

The principle of operation of the BWO is based on the interaction between an electron beam, which is generated by an electron gun, and a slow-wave structure. The excited microwave wave is traveling backward against the beam. When it reaches the electron gun region, it is coupled out by the output port. The BWOs are considered as very stable and powerful microwave sources, although the maximum output power level for O-type is in a range up to 1 mW at 1000 GHz. Nevertheless, the good quality waveform they produce makes them very good illuminators in terahertz imaging and EPR spectroscopy. The BWOs are widely used in EPR spectroscopy, for example a set of four BWOs were used in [24] in so called high frequency EPR (HFEPR), where high fields are employed in combination with multiple energy sources in the sub-THz range.

Another example of using the BWO as microwave sources can be found in [25], where results of studying the structure of the paramagnetic centres formed by impurity Ho^{3+} ions in synthetic forsterite (Mg_2SiO_4) are presented. They used a basic set of BWOs which permit operation in a frequency range of 60 to 1500 GHz [26]. Unfortunately, the output power of the BWO at frequencies over 100 GHz are at milliwatt level. This factor limits usage of the BWOs at higher frequencies and one needs to use other microwave sources.

2.6. Orotron

The orotron was proposed in 1969 by the Soviet physicists F. S. Rusin and G. D. Bogomlov [27]. It consists of an open resonator (Fig. 3) with two mirrors, one of which has a periodic structure. The electron beam is located in a focusing magnetic field directed parallel to the motion of the electrons. The periodic structure produces the slow spatial harmonics of the cavity field. Electrons interact with one of such harmonics under the condition:

$$\omega \approx h\nu, \quad (3)$$

and then a simple relation between the radiation wavelength λ and structure period d can be used:

$$\lambda \approx \frac{d}{\beta}, \quad (4)$$

where ω is the angular frequency of the cavity mode, $h = \frac{2\pi}{d}$ is the longitudinal wavenumber of the slow spatial harmonic of the field, ν is the electron velocity, $\beta = \frac{\nu}{c}$ and c is the speed of light.

Orotrons are currently being developed for generating high frequency radiation [28], [29] of up to 410 GHz at a 50–200 mW power level. Nevertheless, the efficiency of this device is quite low due to the large number of periods in the periodic structure and the non-uniformity of the microwave field magnitude at the electron beam cross section.

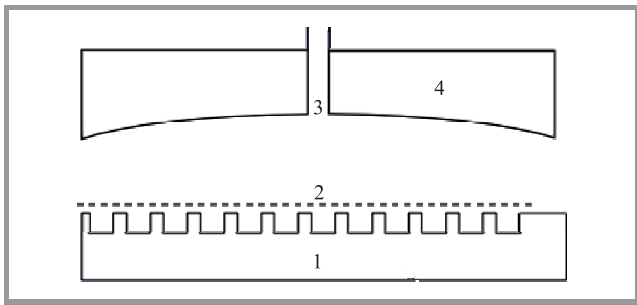


Fig. 3. Structure of an orotron: 1 – mirror with periodic structure, 2 – electron flux, 3 – radio wave guide, 4 – mirror.

Efficiency does not usually exceed 0.1–1% at submillimeter wavelengths [30].

In contrast to the gyrotron, the orotron is a smaller and more compact device. It also does not require a high intensity of the magnetic field. What is more, it does not require a complicated power supply and cooling systems. Therefore, the output power levels generated by the orotron are several orders of magnitude less than those generated by the gyrotron.

2.7. IMPATT Diode

The IMPATT diode (IMPact ionization Avalanche Transit-Time diode) is a powerful semiconductor device used to produce millimetre waves and microwaves. Its structure, similar to the basic PN junction, was first proposed by Shockley (1954) [31] and further developed by Read (1958) [32].

Transit time of the carriers in the junction results in the negative resistance effect, which is responsible for the generation of the RF signal. IMPATT diodes cover the frequency range from X-band (6 GHz) to 400 GHz. Their CW power was reported at 12 W at 6 GHz, 1 W at 94 GHz [33] and 2.2 mW at 412 GHz [34].

These kind of generators are widely used in EPR spectroscopy, especially in the former Soviet Union where this technology was highly developed [35].

The main advantage of IMPATT diodes is their noise-performance, which is comparable to klystrons. What is more, the price is much lower, the lifetime is long and only a simple power supply is required [36]. The only drawback of the IMPATT diodes is a high level of phase noise, which is not desirable at EPR spectrometer setup. Considering all disadvantages and advantages the IMPATT diodes can be very promising microwave source, but one needs to use proper methods to compensate the phase noise.

2.8. Gyrotron

The devices that are widely used as a microwave source in EPR/NMR experiments are gyrotrons [37], [38]. The gyrotron was invented in the Radiophysics Institute in Gorky (now the Applied Physics Institute in Nizhny Novgorod) in the 1960s [39]. The gyrotron is a kind of maser (strictly speaking it is a cyclotron resonance maser – CRM), which

is equivalent to a laser for microwave wavelength, and as with the laser, the gyrotron is also an oscillator. This device is used with success in many experiments, where a stable, high frequency and high power microwave source is needed. The highest frequency reached by the gyrotron is now about 680 GHz [40], with an output power level reaching about 300 kW.

Despite the main application for gyrotrons being plasma heating in tokamak installations, they have attracted the attention of many researchers that are conducting experiments involving various kinds of spectroscopy. For the last few years NMR/DNP [41] spectroscopy experiments have been widely reported. The first commercial NMR/DNP spectrometers are available. There is continuous research towards higher radiation frequencies, which was recently reported during a workshop at University of Fukui, a leading centre in gyrotron research [42].

During the above-mentioned workshop in Fukui, the latest research results in the field of ESR spectroscopy, which involved the use of the gyrotron, were presented [43]. The experiments were conducted with the use of the gyrotron, which operated at a frequency of 154 GHz and with an output power of 150 W. The use of this kind of system will allow for further development of material engineering, especially in the context of materials with a short relaxation time. Similar research is being held in many other institutions e.g. in MIT [14], where the gyrotron for use in NMR spectroscopy was designed and built. It operates at a frequency of 460 GHz with a maximal output power of 100 W.

The gyrotron, as an electromagnetic radiation source, is a very promising device, mainly due to its simple construction, high efficiency and potential possibility to achieve frequencies of up to several terahertz. The development of the gyrotron technique and technology is in the scope of interest of several countries, including Poland [44].

Frequency and power stability in the gyrotron has been studied for some time. MIT has been conducting experiments with a tunable gyrotron for spectroscopy applications since 1990 [45]. There is worldwide interest in this type of device. The gyrotron reported in [45] has a long term power stability of 0.7% at 2.71 W of power and ± 6 ppm frequency stability at 460 GHz. On the other hand, the gyrotron reported in 2002 [41] achieved a power stability of 1%. It is quite an improvement as the gyrotron reported in 1994 was operating at a frequency of 95 GHz with a frequency stability of 100 ppm [46]. The same group from Fukui, Japan reported in 2010 a wide range (1.5 GHz) continuous tunable gyrotron working at 394.6 ± 0.6 GHz [37]. Unfortunately in this work, no stability test results nor measurement sensitivity gain have been reported.

3. Summary

The most important microwave sources were briefly discussed and the need of new microwave sources and the development of existing ones has been pointed out.

Before some advice will be given on choosing the proper microwave oscillator for use in EPR, let once more time revise the reason why one need to use higher frequency. There are several advantages of a higher frequency source in EPR applications [24]:

1. Second-order effects are reduced at high magnetic fields. Higher radiation frequency allows the use of the higher magnetic field.
2. A higher magnetic field and higher operation frequency increases spin splitting, thus spectral resolution over the g factor is higher. Better resolution is important in the investigation of polarity, structure and spin.
3. Exponential dependency of the number of excited spins on the radiation frequency causes saturation of paramagnetic centers. This dependency is used to study the relaxation and dynamics of the paramagnetic centers. Moreover, cross-relaxation of the paramagnetic centers decreases rapidly at high magnetic fields. As a result it is possible to obtain more precise and complete information about the system under study.
4. Large microwave quantum energy makes it possible to investigate systems with large zero field splitting.
5. Precision of pulse methods also increases at high magnetic fields.
6. Higher orientation selectivity and sensitivity in the investigation of disordered systems.

The choice of the microwave source for the given application must meet all requirements and other specific criteria. Every oscillator has its advantages and disadvantages, and this must be carefully considered.

The most important among them, in the context of improving sensitivity and also measurement dynamics and the further development of spectroscopy, are the gyrotron and orotron.

The gyrotron, because of its high efficiency (up to 50%) and also its high power and ability to generate a continuous wave of high frequency, will allow samples during a long exposure time in constant radiation conditions to be investigated.

But the gyrotron is very expensive and troublesome device and even though it has many advantages, in experiments required frequencies up to 100 GHz can be used other microwave source. The gyrotron is the only choice when one need to operate at higher frequencies, but then much more powerful magnetic field is also needed.

The orotron, because of its compact size and also its pulsed operation and lack of complicated gear (power supply, magnets, cooling system), will allow samples to be even quickly examined in field conditions.

Orotrons can be used as an alternative to the other microwave tubes as well as semiconductor diodes. Orotron can provide higher power levels than diodes and other tubes,

so it can be used as the intermediate source between oscillators of lower frequencies with higher output power and those with higher frequencies and high output power, i.e. gyrotron.

When one need to operate at low frequency the proper choice is gun/IMPATT diodes and cheap vacuum tubes such as TWTs, magnetrons and klystrons.

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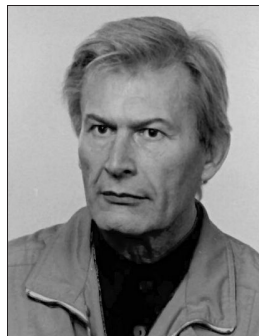


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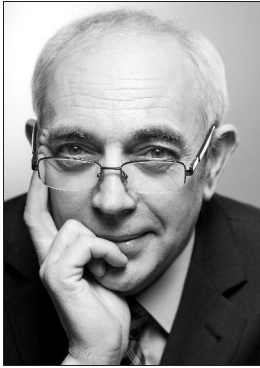
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