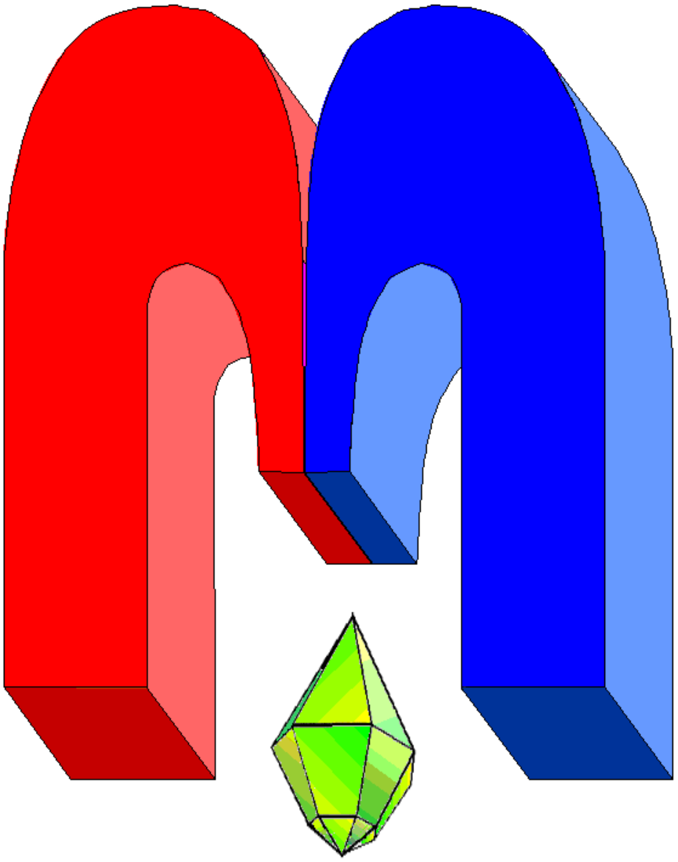
ISSN 2072-5981
doi: 10.26907/mrsej



Magnetic Resonance in Solids

Electronic Journal

Volume ***23***
Issue ***2***
Article No ***21201***
1-6 pages
2021
doi: **10.26907/mrsej-21201**

http://mrsej.kpfu.ru
http://mrsej.ksu.ru

Established and published by Kazan University[*]
Endorsed by International Society of Magnetic Resonance (ISMAR)
Registered by Russian Federation Committee on Press (#015140),
August 2, 1996
First Issue appeared on July 25, 1997

"***Magnetic Resonance in Solids. Electronic Journal***" (**MRS*ej***) is a peer-reviewed, all electronic journal, publishing articles which meet the highest standards of scientific quality in the field of basic research of a magnetic resonance in solids and related phenomena.

Indexed and abstracted by
Web of Science (*ESCI*, Clarivate Analytics, from 2015),
Scopus (Elsevier, from 2012), *RusIndexSC* (*eLibrary*, from 2006), *Google Scholar*,
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[†] In Kazan University the Electron Paramagnetic Resonance (EPR) was discovered by Zavoisky E.K. in 1944.

Multipurpose Portable Q-Band Bridge

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(Received October 1, 2021; revised October 24, 2021;
accepted October 26, 2021; published October 29, 2021)

The paper describes the design, implementation, and technical characteristics of a portable Q-band microwave (mw) bridge based on the Gunn diode with the potential use for electron paramagnetic resonance (EPR) and dynamic nuclear polarization (DNP) spectroscopies. The mw frequency can be electronically adjusted in the range of 36–38 GHz with the maximal mw output of 120 mW and electronic attenuation of 0–60 dB. The value of the mw frequency can be stabilized and changed via automatic frequency control for direct and alternating current. A self-written Matlab-based program allows tuning and operating the bridge through the RS-485 interface. Examples of the EPR spectra implemented into the magnetic system of the Bruker ESP300 commercial spectrometer are shown.

PACS: 76.30.-v, 76.60.-k, 76.60.Lz, 07.57.Pt, 75.78.-n, 89.30.aj

Keywords: microwave; spectrometer; electron paramagnetic resonance; dynamic nuclear polarization

1. Introduction

High initial and maintenance costs, immobility, excess complexity of the commercial Q-band spectrometers for electron paramagnetic resonance (EPR) and microwave (mw) equipment operating at the mw frequency in the range of 34–42 GHz, as well as closed program code for device control, monopoly of manufacture(s) with various sanctions restrictions, forced us to construct portable Q-band bridge for potential use as (a) a mw source to study the effect of mw radiation on the properties of substances and materials in laboratory conditions [1–4]; (b) a mw source for dynamic nuclear polarization (DNP) [5–7]; (c) a necessary part of the EPR spectrometer. There are many reasons for choosing Q-band for the mentioned purposes. Firstly, dielectric losses of water are maximal at Q-band [8] favoring selectivity for the water heating in the study of microwave influence on the water-containing materials. Secondly, as concerning EPR and DNP applications, besides the higher spectral resolution comparing to the conventional for EPR X-band frequencies of 9–10 GHz, the most Q-band EPR measurements can be done by exploiting magnetic systems based on the standard electromagnets while for the higher allowed mw frequencies use of expensive superconducting systems is required [9, 10].

The purpose of this work was to create a portable Q-Band bridge with the potential use as a part of EPR and DNP spectrometers as well as a tunable source of mw irradiation at various locations.

2. Materials and methods

Q-band microwave bridge

The microwave bridge is built according to the classical scheme to obtain EPR spectra (Figure 1). The manufactured according to this block diagram microwave bridge (a box with the size of $60 \times 40 \times 20\,\mathrm{cm}^3$ with a mass of about 3 kg) is shown in Figure 2. 8 mW microwave radiation generated by Gunn diode (1) is amplified to the 120 mW power at solid-state microwave

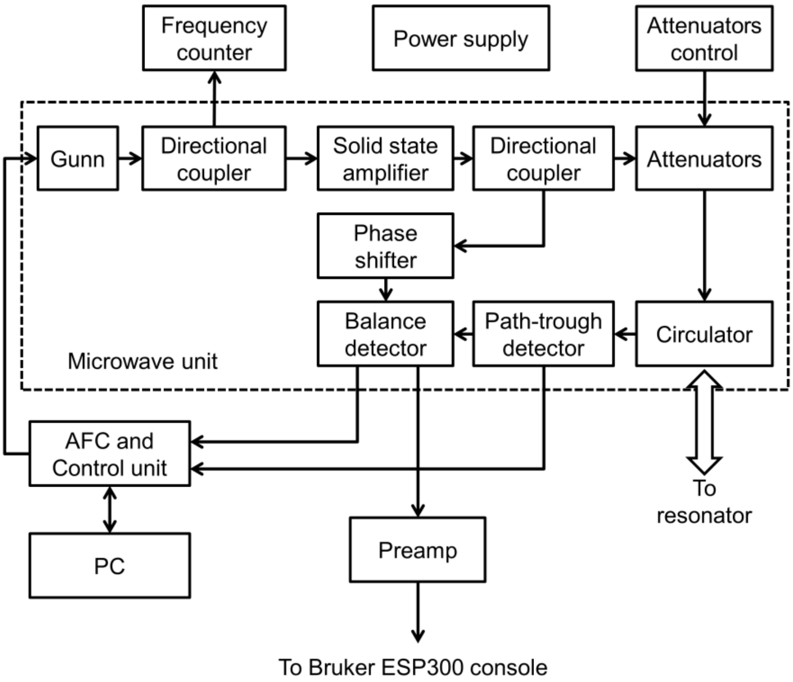


Figure 1. Block diagram of the microwave bridge. For an explanation of abbreviations, see the text

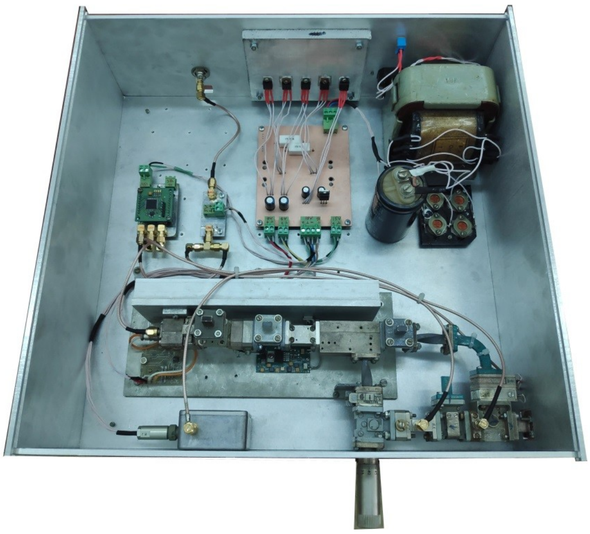


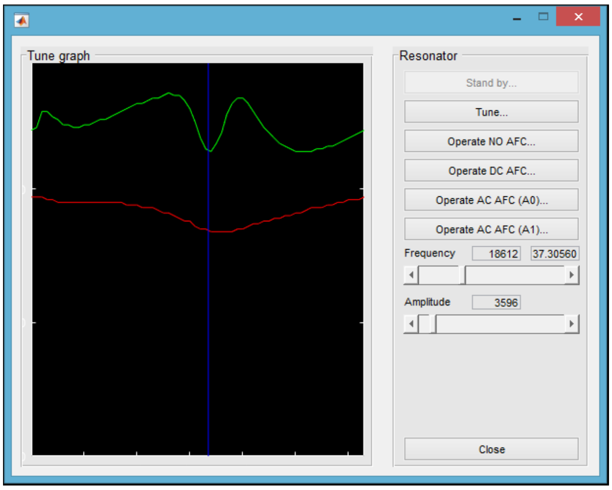
Figure 2. Microwave Q-band bridge with open top covert

amplifier (2). Part of the radiation branched at precise directional coupler (3) then passes two electrically controlled attenuators (4, 30 dB each) and via circulator (5) goes to the resonator. The reflected microwave radiation again via circulator detects first by the path-trough detector (6) and then follows to the balance detector (7). The reference signal branched at directional coupler (3) passes through a phase shifter (8) again to the balance detector (7). Apart from the mw part, the microwave bridge consists of the power supply (9) which together with the specialized power supply of the Gunn (13) and solid-state microwave amplifier (14) provide power for all the necessary parts of the microwave bridge. Attenuation of the microwave attenuators (4) is controlled by the precision current source (10) which provides a direct current (DC) 0–100 mA to the attenuators. The microwave frequency of the Gunn source is stabilized to the frequency of the resonator using a digital automatic frequency control (AFC) board (11). There are three

Table 1. Microwave bridge characteristics

Parameter	**Value**
operating frequency (average)	37 GHz
microwave frequency band	36–38 GHz
maximal output power (defined by solid state microwave amplifier)	120 mW
power adjustment range	0–60 dB
modes of frequency stabilization	without AFC, DC mode, two AC modes
frequency band of the IF signal	500 MHz
control interface	RS-485 with MODBUS RTU protocol

possible modes of mw frequency stabilization: no stabilization, DC stabilization, and alternating current (AC) stabilization. Besides frequency stabilization, this board controls all the parts of the microwave bridge and provides a connection with a personal computer using MODBUS-RTU protocol with an RS-485 physical layer interface. The signal reflected from the resonator after balance detection amplifies at signal preamplifier (12). The resulting low-frequency signal is directed to the Bruker ESP300 console for final synchronous detection and digitizing. The mw bridge characteristics are gathered in Table 1.

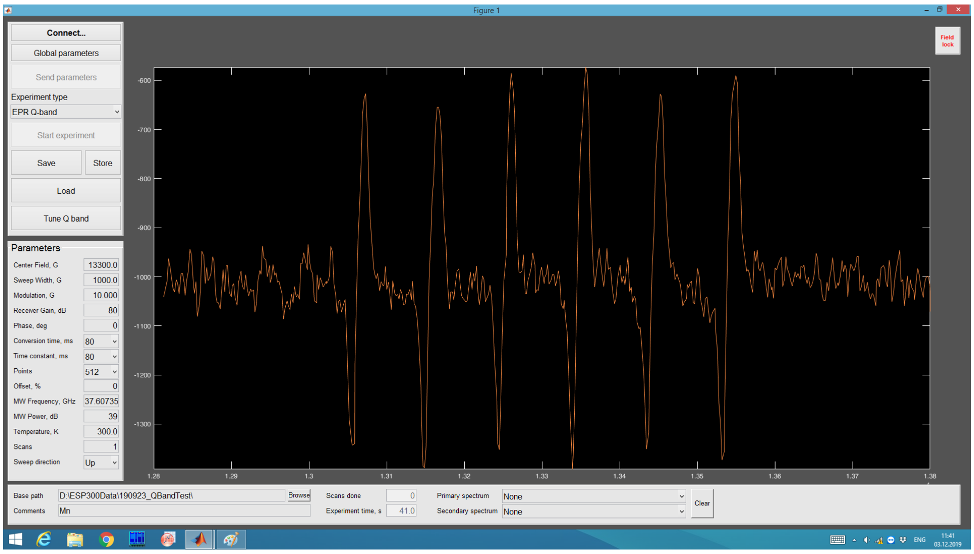


Frequency 18612 37.30560

Amplitude 3596

Figure 3. Program menu item for tuning the microwave bridge with the resonator

The microwave bridge is controlled using control commands from a personal computer via the RS-485 interface using the MODBUS RTU protocol. The control software (written in Matlab) is integrated into the modified program of the Bruker ESP300 spectrometer. In it, you can select the operating mode of the microwave bridge, adjust the resonant frequency, select the operating mode of the autotuning of the resonant frequency (Figure 3). In addition, for



Experiment type: EPR Q-band

Parameters

Center Field, G	13300.0
Sweep Width, G	1000.0
Modulation, G	10.000
Receiver Gain, dB	80
Phase, deg	0
Conversion time, ms	80
Time constant, ms	80
Points	512
Offset, %	0
MW Frequency, GHz	37.60735
MW Power, dB	39
Temperature, K	300.0
Scans	1
Sweep direction	Up

Base path: D:\ESP300Data\190923_QBandTest\
Comments: Mn
Scans done: 0
Experiment time, s: 41.0
Primary spectrum: None
Secondary spectrum: None

Figure 4. Program dialog box for the obtaining of EPR spectrum on the example of Mn^{2+} in MgO

convenient tuning, one can select the frequency scan range. The dialog box of the developed program for measuring EPR spectra using the example of the spectrum of manganese in MgO powder is shown in Figure 4.

3. EPR tests

To test the microwave bridge, a cylindrical resonator with main modes $TE_{01\mathrm{x}}$ was produced, where x = 1,2,3 (Figure 5). Its properties are gathered in Table 2. Obtained characteristics are worse than the properties of the modern Q-band resonators and should be significantly optimized. However, test measurements of the several samples allowed us to estimate some of the microwave bridge characteristics.

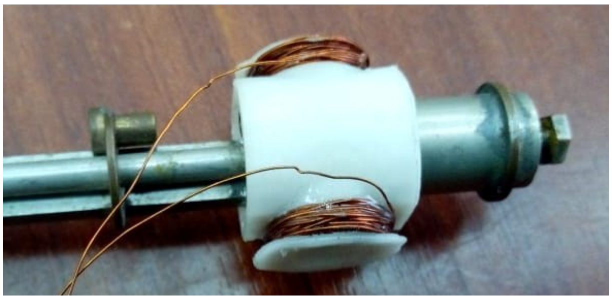


Figure 5. Microwave resonator with modulation coils for observing EPR signals

The EPR spectrum of the manganese doped MgO obtained using above mentioned bundle (bridge and resonator) is shown in Figure 6. The detected six-line pattern represents standard hyperfine interaction of the manganese paramagnetic center with its nucleus (^{55}Mn isotope with the nuclear spin of $I = 5/2$ [11]). The signal-to-noise ratio (SNR) in the spectrum is relatively small and mostly related to the low quality factor of the test resonator. It is known that increase of the magnetic field amplitude modulation leads to increase SNR for the broad lines. We estimated maximum magnetic field modulation amplitude of the test resonator using ultra-narrow signal from sealed in the tube lithium phthalocyanine (LiPc) samples as the reference (Fig. 7). LiPc is widely known material for the EPR oximetry; its linewidth (which can be as narrow as 10 μT in a vacuum) nearly linear depends on the partial pressure of oxygen [12, 13].

Table 2. Microwave resonator characteristics

Parameter	**Value**
operating frequency band of $TE_{01\mathrm{x}}$ modes, where x=1,2,3	35–41 GHz
the possibility of tuning the resonant frequency	yes
the ability to change the matching of the resonator and the microwave bridge	yes
Q-factor of the resonator	100–150 (depends on the mode)
magnetic field modulation on the sample	0–2 mT
maximal sample size	up to 3 mm in diameter

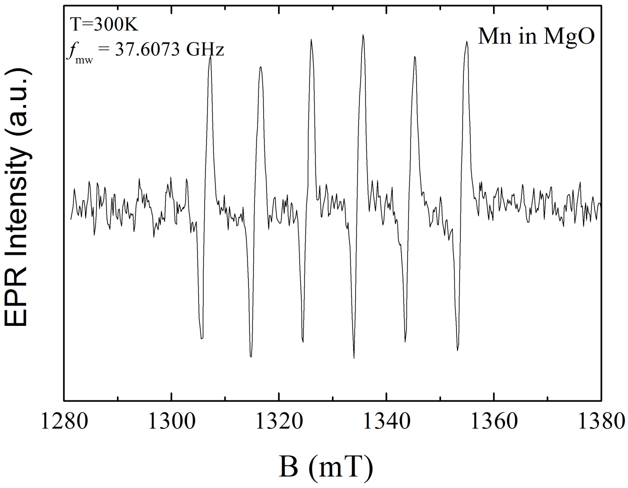


Figure 6. Central part of the EPR spectrum of Mn in MgO acquired at $P_{\mathrm{mw}} = 10\,\mu$W and modulation amplitude of 0.1 mT

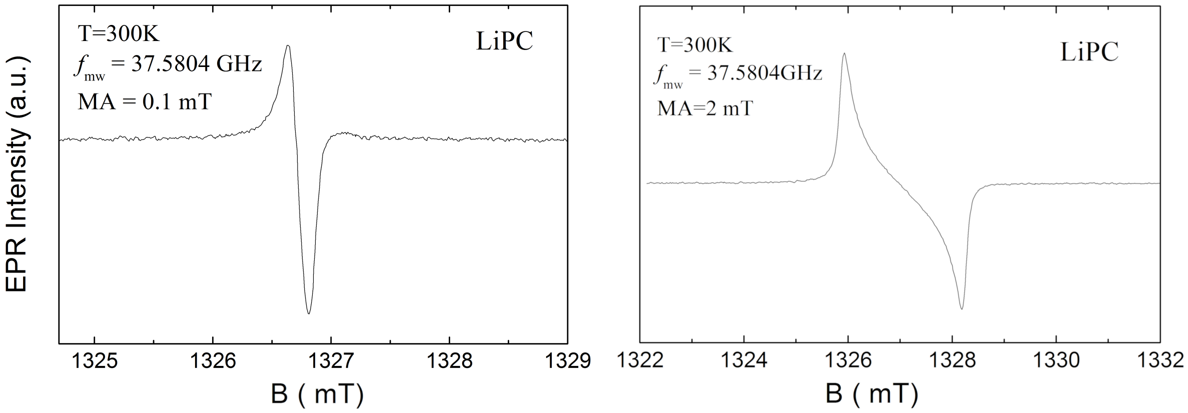


Figure 7. EPR of LiPc acquired at $P_{\mathrm{mw}} = 25\,\mu$W and modulation amplitudes (MA) of 0.1 mT (left) and 2 mT (right)

The left part of the figure 7 represents EPR spectrum of LiPc with modulation amplitude 0.1 mT and the right part corresponds to the highest possible modulation of 2 mT. The nonideal lineshape of the EPR line is due to the saturation of the LiPc signals. Since test resonator does not have the ability of the coupling change, the working point of the detector is tuned by the microwave power level.

4. Conclusion

As it was stated above, main goal of the current work is developing of the low cost microwave bridge for EPR and DNP investigations. Technical characteristics of the bridge are quite promising and similar to those of the commercially available cw bridge. Unfortunately, absence of the proper resonator did not allow us to estimate real parameters of the bridge itself and we restrict ourselves to the rough experiments with measurements of the relatively strong signals. Thus, the next stage will be the development of the low cost resonator cavity, which will be a supplement for the developed Q-band microwave bridge.

Acknowledgments

The work is financially supported by Russian Science Foundation (grant #19–12–00332)

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