

ESR SPECTROSCOPY EMPLOYING YIG-TUNED OSCILLATOR IN CONJUNCTION WITH LOW-QUALITY FACTOR RESONATORS

Lazar Shtirberg, Michael Shklyar, and Aharon Blank
Schulich Faculty of Chemistry, Technion — Israel Institute of Technology, Haifa 32000, Israel
E-mail: lazarah@tx.technion.ac.il

Abstract — Electron spin resonance (ESR) is a very useful and robust microwave spectroscopic method with many applications in science and technology. Common microwave sources used in ESR spectroscopy, such as klystrons or Gunn diodes have small bandwidth of ~1GHz (at 10 GHz) and need to be locked on high-Q cavity ($Q > 1000$) to achieve low phase noise at 100 kHz offset. Here we developed an experimental system with YIG-Tuned Oscillator, which can be used for ESR spectroscopy in the entire range of 8-18GHz and can be locked on relatively low Q resonators ($Q > 200$). The hysteresis of YIG-Tuned Oscillator does not affects its performance and even at the low Q values one obtains tracking error of the resonator frequency that are ~10-100 kHz.

I. Description of the System

The experimental system with the YIG-Tuned Oscillator is shown in Fig.1.

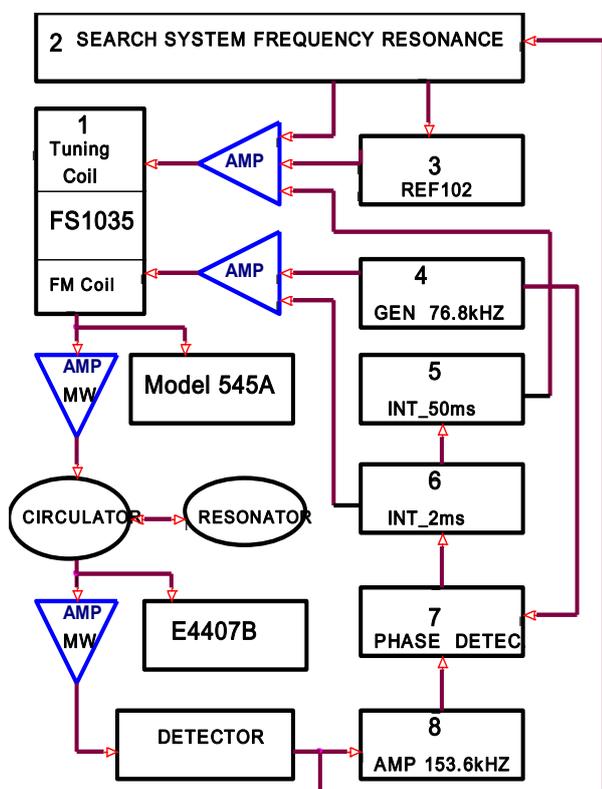


Fig. 1. The ESR system with the YIG-Tuned Oscillator

The aim of this system is to lock the microwave frequency on the resonance frequency of the resonator (so called automatic frequency control - AFC) that may drift due to temperature changes or other system variations: The microwave signal originates in the YIG-Tuned Oscillator (1), model FS1035 from Teledyn, with a tuning range of 8-18 GHz, hysteresis of 10MHz, typical phase noise of -125dBc/Hz at 100KHz; tuning coil sensitivity of 20 MHz/mA, and FM coil sensitivity of 400 kHz/mA (with total deviation range of 40MHz). The frequency of the oscillator can be monitored for test purposes by an EIP 545A frequency counter. The oscillator output is ampli-

fied; goes through a circulator to the resonator and the reflected signal can be measured by an Agilent E4407B spectrum analyzer (for test purposes), or amplified and detected by a simple diode detector. The signal from the diode detector is used for the AFC correction.

The center frequency of operation is determined by the output of AMP #1. This amplifier receives its primary input from module (3), which is constructed of a precision 10V voltage reference source (REF102) with a drift of 2.5ppm/°C and noise of 5 μ Vp-p at the 0.1Hz-10Hz bandwidth range. The voltage from (3) determines the center frequency of operation of the system, and can be adjusted manually. Module (2) is another input for AMP #1 and is used during the "tune" mode of the system to sweep fast the frequency of the oscillator and observe the reflection coefficient of the resonator as a function of frequency. The third input of AMP #1 corrects for frequency shifts of the resonator, as will be detailed below.

In our experiments we employ a dielectric resonator whose resonance frequency greatly depend upon the temperature [1]. The resonator is made of monocrystalline rutile (TiO₂) [2]. The drift is 2.5MHz / °C.

Our measurements involve the use of gradient coils around the resonator that are operated at relatively high power and heat-up their environment. As a result of that the resonator temperature can change in 10 and even 20 °C, leading to resonance frequency changes of up to ~200 MHz. The AFC functionality that compensates for these large changes is achieved through the commonly used method of oscillator frequency modulation [3] that is adapted here for the uncommon case of large frequency variations of and low Q resonator, as shall now be described:

The YIG source frequency modulation is achieved by the use of the FM coil that gets a 76.8kHz input from module (4) through AMP #2. When the average frequency of the source is exactly on resonance, the reflected power, as detected by the diode, should correspond to the second harmonics of the modulation signal, i.e., 153.6 kHz. This signal is amplified in module (8). The symmetry of the signal after the phase detector (7) determines the accuracy of the settings. After the phase detector the signal is integrated with a 2 ms integration window (6) and the error signal is fed to the FM Coil through AMP #2.

Fig. 2 shows the current compensation for the FM Coil (IFM) during typical operation, and Fig. 3 shows the spectrum of this current. As seen in Fig. 3, the main problems in stabilizing the frequency of the YIG source are at frequencies of 150 Hz, 50 Hz and their harmonics. The device uses a DC power supply with single phase input from the grid. However, the three-phase contribution also affects the device. The shield we use for the electronics is made of 2 mm thick aluminum, which does not protect against these effects. Based on these measurements, the calculated variations in the tuning coil input voltage at 150 Hz are ~18 μ V.

While the FM coil compensates for short term fluctuations in the source frequency, it has limited scan range and thus slower but potentially large frequency

drifts are compensated through the main tuning coil. This is achieved by the input to AMP #1 coming from the 50 ms integration module (5).

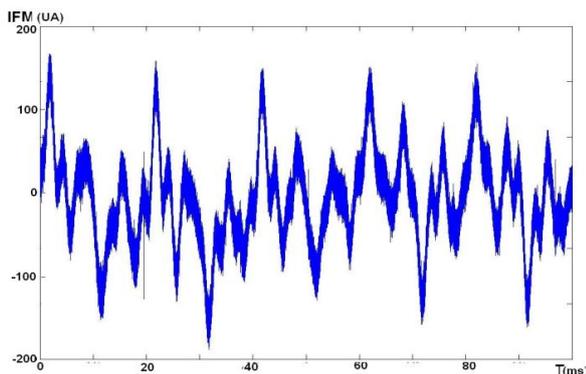


Fig. 2 The current compensation of FM Coil (IFM)

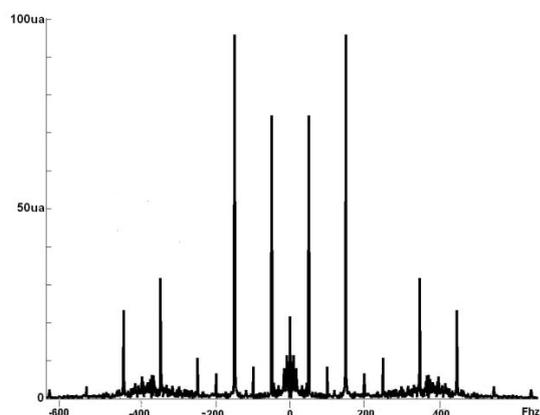


Fig. 3 The spectrum of current compensation of FM Coil

II. Examples of System Operation with YIG Oscillator

In order to evaluate the performance of the system and the relatively long time scale correction method, the frequency of the source was monitored in 1s intervals by the IIP frequency meter. Fig.4 shows typical results of the time dependence of the frequency of the YIG oscillator when working with resonator with $Q = 300-350$, at a frequency around 17.1265 GHz. The temperature in this case was relatively stable so the tuning coil long time correction was not employed. Small thermal fluctuations of $\sim 0.1^\circ\text{C}$ cause the resonance frequency to shift in the range of 1 MHz. The relatively low Q of the resonator leads to relatively large frequency fluctuations, mainly due to non-symmetric reflection coefficient near the resonance frequency. As a result, the accuracy of retention is around 50 kHz.

Fig. 5 shows the dependence of the frequency of YIG oscillator while working with a different resonator at 16.7715 GHz, having $Q = 300-350$. The graph was recorded during an actual ESR imaging experiment, where the resonator temperature varies significantly. The current in the gradient coils is turned "on" and "off", thus changing the temperature and this results in the "saw tooth" form in the resonance frequency.

The resonance frequency increases with increasing temperature. It was found that during the cooling down period, after the experiment ended, cavity is more stable. During two minutes of operation the shift was more than 50MHz

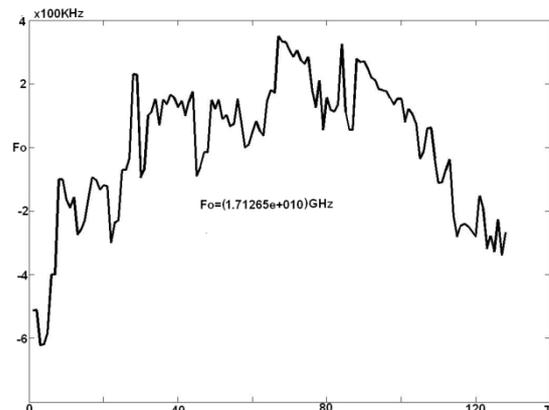


Fig. 4. A plot of the frequency of YIG oscillator while working with $F_0 = 17.1265\text{GHz}$; $Q = 300-350$

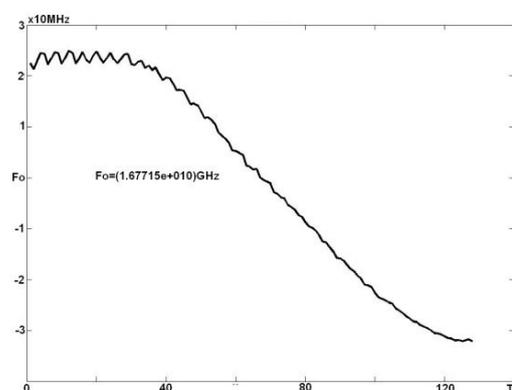


Fig. 5 A plot of the frequency of YIG oscillator while working with $F_0 = 16.7715\text{GHz}$; $Q = 300-350$

III. Conclusions

A wide band ESR microwave system based on a YIG-Tuned oscillator with AFC module was presented. The system was successfully used for ESR spectroscopy and ESR imaging experiments. Tracking error between the oscillator and the resonator is in the 10-100 kHz range. The possibility to work at several frequencies with a single system can significantly expand the possibilities for continuous wave ESR spectroscopy.

IV. References

1. Blank, A., Halevy, R., Shklyar, M., Shtirberg, L. and Kuppusamy, P., "ESR Micro-Imaging of LiNc-BuO Crystals in PDMS: Spatial and Spectral Grain Distribution," *Journal of Magnetic Resonance*, 203 (2010) 150-155.
2. Michael Edmund Tobar, Jerzy Krupka, et al. Anisotropic complex permittivity measurements of mono-crystalline rutile between 10 and 300 K. *Journal of Applied Physics / Volume 83 / (77-79) October 1997.*
3. Charles P. Poole, "Electron Spin Resonance: A Comprehensive Treatise on Experimental Techniques," Courier Dover Publications, 1982.