

A Single-Chip Dual-Mode CW/Pulse Electron Paramagnetic Resonance Spectrometer in 0.13 μm SiGe BiCMOS

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Abstract — Microwave circuitry for electron paramagnetic resonance (EPR) spectroscopy is implemented in a 0.13 μm SiGe BiCMOS process. The chip can operate in both continuous wave (CW) and pulse modes. The frequency is tunable from 770MHz to 970MHz, corresponding to Zeeman magnetic fields from 28mT to 35mT for a free electron. The CW-EPR absorption line of a DPPH powder sample is acquired. The chip consists of a VCO, a PA, an LNA, a down-conversion mixer, baseband amplifiers, and a pulse generation block.

Index Terms — Integrated circuits, Spectroscopy, Silicon germanium.

I. INTRODUCTION

Electron paramagnetic resonance (EPR) phenomenon is based on interaction of electron spins with electromagnetic fields in the presence of an external DC magnetic field [1]. EPR data provides valuable information about electronic structures and spin interactions in paramagnetic materials. EPR has found wide-ranging applications in science, engineering and medicine. Examples of applications are: determining protein folding structures, studying lattice defects in crystals, studying chemical reactions involving free radicals or transition metal ions, performing ionizing radiation dosimetry both for calibration purposes and *in vivo* dosimetry (e.g. detection of radiation-induced free radicals trapped in human bone, tooth, etc.). In medical research, EPR spectroscopy and imaging techniques are employed for investigating tissue oxygenation levels, free radical generation in cancer, and cardiovascular diseases. Reference [2] provides a general review of EPR applications.

Conventionally, EPR spectrometers comprise of components that are expensive, heavy, and large in weight and physical dimensions. Integration of EPR circuitry in a single chip is a step toward in making EPR less costly and more portable.

In this paper, for the first time, a dual mode EPR spectrometer in a conventional silicon process is reported that is capable of performing both continuous wave (CW) and time-domain (pulse) measurements. The complete system is tested and verified by performing CW-EPR measurement for 2,2-Diphenyl-1-Picrylhydrazyl (DPPH) powder at room temperature. The measurement results match closely with theoretical results, illustrating successful functioning of the spectrometer.

II. EPR BACKGROUND

The main components of the presented EPR spectrometer in this paper include a single-chip transceiver, a magnet with adjustable magnetic field, and a planar microstrip parallel loop-gap resonator. The block diagram of the spectrometer is presented in Fig. 1, where the on-chip transceiver is shown in the dashed section. The sample and the resonator are placed inside the magnet that generates a DC magnetic field, B_0 , also known as Zeeman field. Presence of this external field introduces an energy difference ΔE between the two spin states of an unpaired electron: parallel and anti-parallel to B_0 , with ΔE being proportional to B_0 . In its resonance frequency, the resonator produces a RF magnetic field B_1 . At the RF frequency where hf equals ΔE (Larmor frequency), where h is the Planck constant, spin transitions between the two up and down spin states occur, resulting in absorption of RF energy in the sample. In a reflection type resonator, this results in a change in the level of reflected power from the resonator.

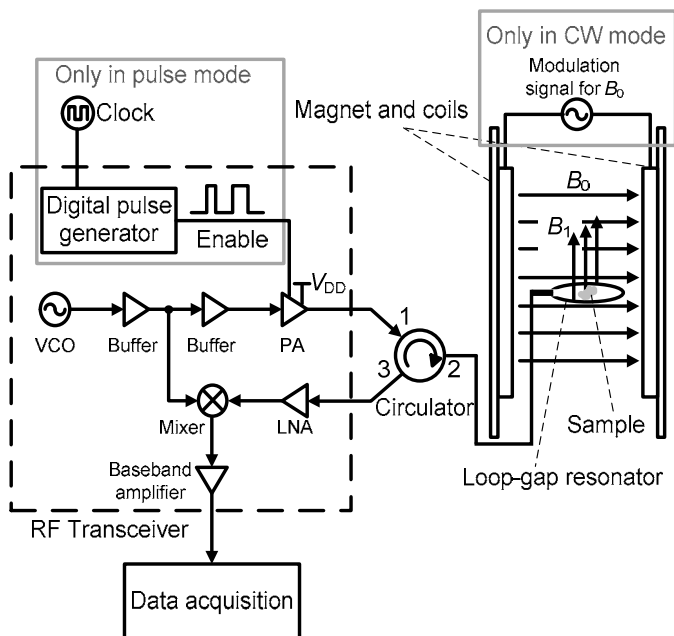


Fig. 1. The block diagram of the EPR spectrometer.

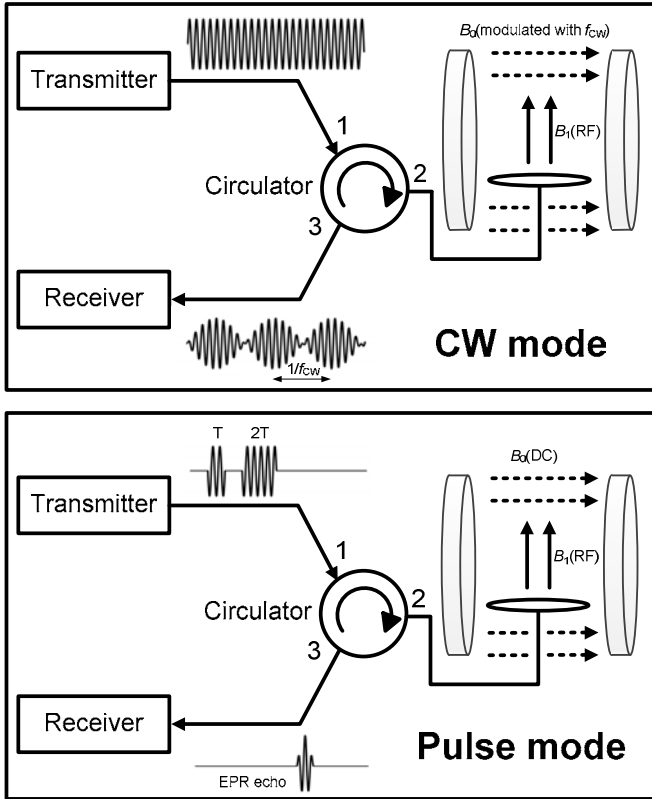


Fig. 2. The operation of the CW and the pulse modes.

Operation principles of CW and pulse EPR modes are illustrated in Fig. 2. A brief description of the two modes of EPR is as following: in both modes, the sample is placed at the center of an RF resonator inside a DC magnetic field, B_0 . In CW-EPR, a continuous RF signal is sent to the resonator and the change in reflected power is monitored to calculate the RF power absorbed by the sample. The absorbed power varies with the B_0 field and this absorption line shape reveals paramagnetic properties of the sample. In order to reduce the effect of low-frequency noise ($1/f$), B_0 is modulated and the reflected power is measured at the modulation frequency.

In pulse-EPR, the transmitter sends a sequence of RF pulses to the resonator, which results in flipping of the spins of the unpaired electrons. The external magnetic field causes precession of the perpendicular or transverse component of the magnetization vector around the B_0 axis. Furthermore, an interaction of electron spins with each other and with nearby nuclei spins results in relaxation of perpendicular (transverse) and parallel (longitudinal) components of the magnetization with respect to B_0 . At the end, the magnetization vector (spin) is aligned with B_0 . The precession of the transverse component generates a time-domain signal detected by the resonator (free induction decay). More complicated echo signals can be generated in studying spin interaction mechanisms. Detailed description of pulse EPR principles and techniques can be found in [3].

III. SYSTEM IMPLEMENTATION AND MEASUREMENT RESULTS

In this work, the integrated EPR spectrometer is tested using $\sim 50\text{mg}$ of 2,2-Diphenyl-1-Picrylhydrazyl (DPPH) powder at room temperature. The Zeeman field is set to around 340G (0.034T), corresponding to 950MHz absorption frequency. This frequency of operation is chosen as a compromise: lower frequencies would result in lower SNR, yet at higher frequencies generating magnetic fields for portable systems would be problematic. The RF signal is generated using an on-chip voltage controlled oscillator (VCO). The VCO, shown in Fig. 3, adopts a differential structure with an LC tank. The VCO frequency can be tuned from 770MHz to 970MHz by changing the bias voltage of the varactors that are placed in parallel with the LC tank. The relationship between the VCO frequency and the tuning voltage is presented in Fig. 4. The VCO signal is amplified by a differential buffer and is converted to a single-ended signal. It is then fed to an on-chip PA for more amplification.

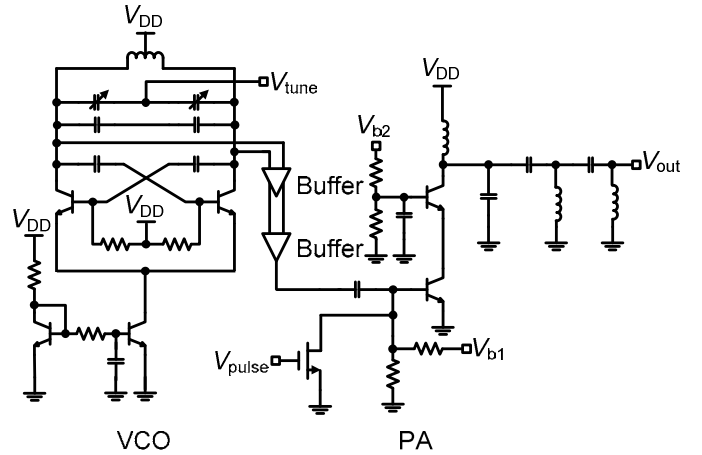


Fig. 3. Circuit topology of the VCO and the PA.

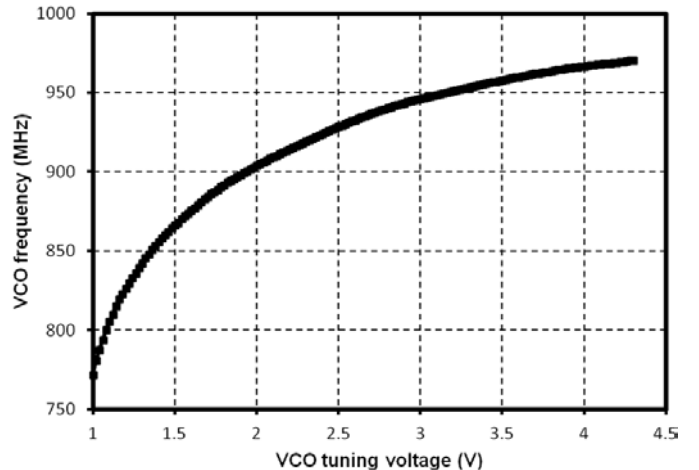


Fig. 4. VCO frequency versus the tuning voltage.

The PA has a cascode topology and provides a maximum power gain of 15dB. The output of the PA is matched to 50Ω by a combination of on-chip inductors and capacitors. The PA output signal is sent to the planar loop-gap resonator via a

circulator. The flat loop-gap resonator is made in-house using a 20mil Rogers 4350B PCB. The loop has inner and outer diameters of 4mm and 5mm, respectively. The loaded quality factor Q of the resonator is measured to be 60. Variable capacitors are applied in parallel and series, to tune the resonance frequency and match the input impedance to 50Ω .

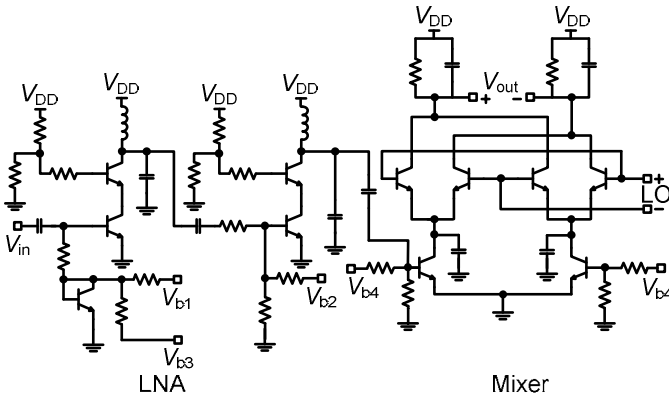


Fig. 5. Circuit topology of the LNA and the mixer.

On the receiving end, a direct-conversion architecture is adopted which consists of a LNA, a mixer, and baseband amplifiers. The reflected RF signal from the sample is coupled to the input of the LNA. This signal is down-converted and its amplitude is measured at the baseband. The LNA has two stages and it is designed to have a power gain of 40dB and a noise figure of 3.6dB at the entire frequency range of the VCO. The designed LNA output 1dB compression point is -15dBm. The mixer has a Gilbert topology with a resistive load. The circuit diagrams for the LNA and the mixer are shown in Fig. 5.

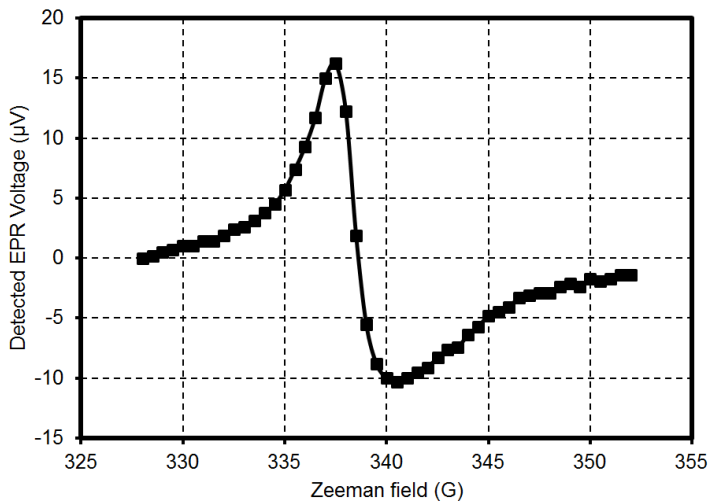


Fig. 6. The measured EPR response of a DPPH sample versus DC magnetic field.

In Fig. 6, a room-temperature CW EPR response of the DPPH sample measured by the single-chip spectrometer is reported. The Zeeman magnetic field B_0 is swept from 328G to 352G. The VCO frequency is kept constant at 950MHz,

which is the resonance frequency of the loop-gap resonator. In order not to saturate the EPR signal and not to introduce significant line broadening, the gain of the buffer and PA in the integrated transmitter is deliberately lowered and a relatively low power of -20dBm is sent. This amount of RF power generates about 25mG magnetic field at the center of the resonator. B_0 is further modulated by a 2kHz signal with 0.27G amplitude to reduce the Flicker ($1/f$) noise. The response curve in Fig. 6 is the first-derivative of the Lorentzian absorption line. There is a separation of 2.5G in B_0 between the positive and negative peaks that includes the broadening effects caused by the field modulation. This matches well with the first harmonic Lorentzian line shape of DPPH powdered crystal [4]. The difference in magnitude of the two peaks comes from the frequency dependency of the phase of the reflected signal. This effect can be minimized by employing an I-Q topology or adding a phase-shifter in the signal path.

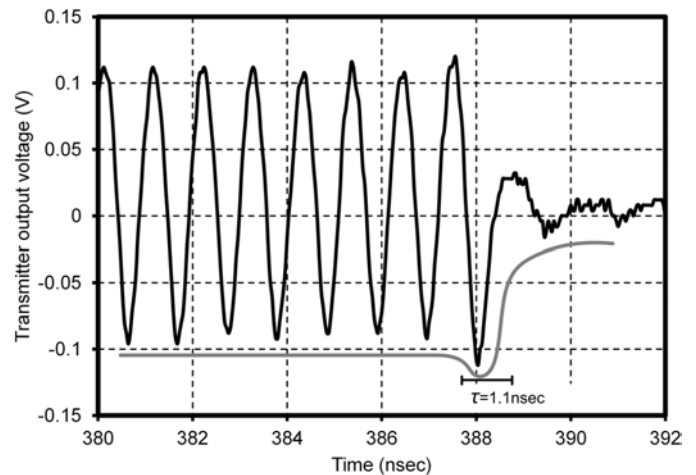


Fig. 7. The output of the transmitter in the pulse mode.

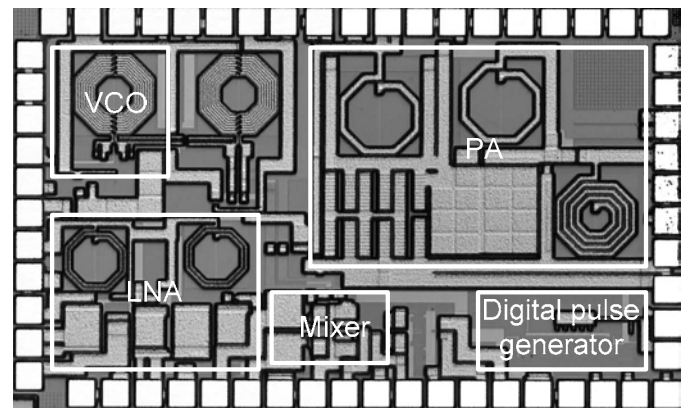


Fig. 8. The micrograph of the chip.

In addition to the CW mode, the chip can be operated in the pulse mode. In this design, a sequence of coherent pulses is generated by an integrated programmable pulse-width modulator. The width of each pulse can be independently varied from 20ns to 400ns with steps of 370ps. The PA can be

turned off by pulling down the base voltage of its input transistor using an NMOS driver. In the pulse mode, the speed that a transmitter can be switched off determines the dead time of the spectrometer. Since the EPR echo is much weaker than the excitation signal, it can only be detected after switching off the transmitter. The amplitude of the EPR echo decays exponentially, and therefore, a small turn-off time is extremely important in the pulse measurements. The measured transmitter output, during the switching process, is shown in Fig. 7. The transmitter can be turned off in less than 1.2ns.

Finally, we present the chip micrograph in Fig. 8. The chip size is 1.5mm by 2.5mm. It is implemented in IBM's 0.13 μ m SiGe BiCMOS process technology.

IV. CONCLUSION

In this paper, we have presented, for the first time, a dual-mode EPR spectrometer in a conventional silicon process that is capable of performing both continuous wave (CW) and time-domain (pulse) measurements. Good matching between measurement and theoretical results demonstrated the functionality of the proposed system.

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