A Novel Probehead for an Electron Spin Echo Study of an Australian Coal

N. Suwuntanasarn[a], W.D. Hutchison[a], G. Milford[b] and R. Bramley[c]

[a] *Centre for Quantum Computer Technology, School of Physical, Environmental and Mathematical Sciences, UNSW@ADFA, Canberra ACT 2600.*
[b] *Centre for Quantum Computer Technology, School of Information Technology and Electrical Engineering, UNSW@ADFA, Canberra ACT 2600.*
[c] *Research School of Chemistry, The Australian National University, Canberra ACT 0200.*

An X-band pulsed electron spin echo (ESE) probe head, which can be fitted with a CW-ESR cryostat, has been designed and tested. The three dimensional electromagnetic field aspects of the structure have been fully assessed and optimised. The theoretical simulations and experimental results agree well. The pulsed ESE system is used to perform detailed ESE studies of a "Queensland coal" sample.

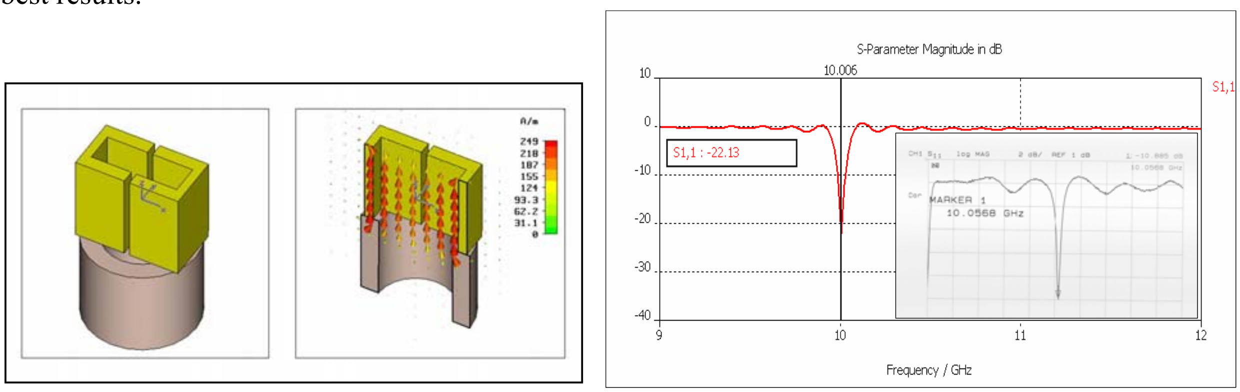
1. Introduction

Pulsed electron spin echo (ESE) is a very powerful tool for studying relaxation phenomena. Here an X-band ESE probe head has been designed specially for a study of phosphorus doped silicon (Si:P) with quantum computing applications in mind [1]. The probe head is designed to fit a conventional CW-ESR cryostat and allow quick sample access at low temperature (to 4 K). The probe head resonator design is based on a loop gap resonator (LGR) structure [2, 3]. In the design, the three dimensional electromagnetic field was simulated numerically using a 3D electromagnetic simulator (CST Microwave Studio [4]). The whole probe head structure was also tested experimentally, and the results are in agreement with the simulations. ESE was then performed on a "Queensland coal" sample in order to test the probe design and our home built pulse spectrometer ahead of Si:P work. A coal sample is a convenient single line sample for testing an ESE system since pulsed ESE studies on various types of coals exist for comparison [5]. Interestingly there appears to be no reported ESE study of Australian coal.

2. Probe head design

A conventional ESR-900 Oxford instrument cryostat shield is replaced by the probe head. The probe head consists of a resonator, microwave and cryostat shield, tuning and matching structure. The shield was designed so that its cut-off frequency is a few gigahertz higher than the resonant frequency to prevent microwave radiation leakage. The structure also incorporates the top vacuum seal for the cryostat. The probe head is adjustable both up-down and in rotation to allow the tuning of the resonator. Microwave coupling into the resonator is via a rectangular iris and Gordon coupler [6], which is rigidly attached to the shield. Matching is achieved by adjusting a movable Teflon insert in the Gordon coupler. Optimisation of the entire probe head structure was carried out using the simulation tool to achieve a good impedance match at the resonant frequency (low S_{11} magnitude, $|S_{11}|$) and high conversion factor ($\Lambda = B_1/(PQ)^{0.5}$, where B_1 is the magnetic induction in the rotating frame at a given microwave frequency, P is the microwave incident power and Q is the quality factor of the resonator).

The resonator is a rectangular LGR shape which is particularly suitable for flat samples (wafers). Samples up to 5 mm width (x-axis), 1.7 mm thick (y-axis) and 5 mm height (z-axis) can be inserted into the resonator. The resonator structure and the simulated magnetic field are shown in Fig.1. A relatively large magnetic field with reasonable homogeneity is produced over the sample area. Measurement was performed with a network analyser and close agreement was obtained between the resonant frequencies and coupling figures (simulated: 10.006 GHz and measured: 10.0568 GHz from the $|S_{11}|$ values) (see the fig.1 (c)). The Q (calculated from the relationship $Q=f/\Delta f$, where Δf is the width of the resonance curve at -3 dB level) of the real structure is approximately 230. The large magnetic field and low Q values produced by the probe head allow a good conversion factor to be obtained and make it particular suitable for a pulsed ESE experiment. With the flat coal sample, the resonator performance was also assessed against other types of the resonator such as folded-half wave and TE_{102} rectangular cavity, and it was found that this resonator gave the best results.



(a) (b) (c)

Fig. 1. The resonator simulated results (colour online): (a) the resonator structure; upper part (yellow) is copper while lower part (grey) is quartz, (b) the corresponding microwave magnetic field (arrows) at the sample position (MWS views) and (c) the simulated and measured (insert) $|S_{11}|$ values.

3. Electron spin echo results

An ESE experiment was performed on "South Blackwater" coal from Queensland with our homemade ESE spectrometer and the resonator described above. The coal sample consists of 85.8% C, 5.07% H, 6.3% ash. The g-factor of the sample, representing electrons bound to free radicals, was found from CW-ESR to be 2.0029, compared to the free electron g-factor (= 2.0023).

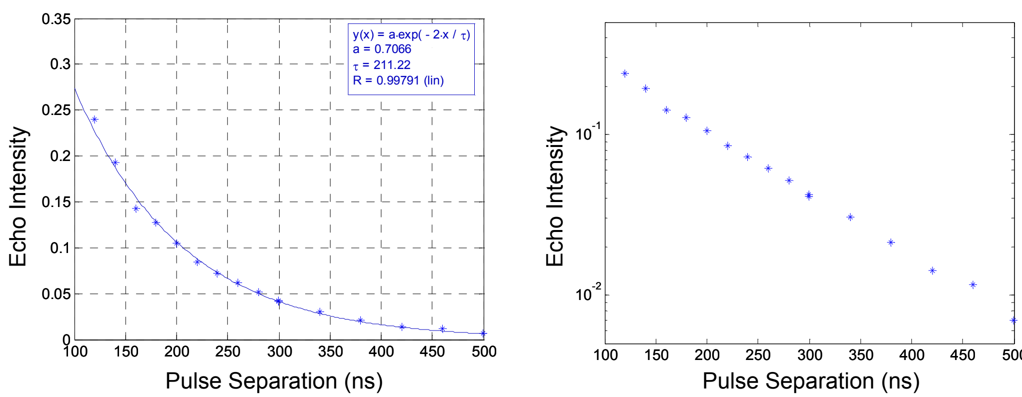


Fig. 2. The echo intensity at various pulse separations, τ, with (left) its corresponding semi-log plot.

A Hahn two-pulse ESE ($\pi/2$-τ-π-echo) was performed initially at varying pulse widths ($\pi/2$ and π) in order to find the optimum pulse width for the experiment. The optimum $\pi/2$ pulse was found to be 22 ns. The spin-spin relaxation time, T_2, of the sample is found from the plot of ESE intensity versus the variation of pulse separation time, τ. The plot of echo intensity versus the pulse separation, both linear and semi-log scale, is in Fig.2.

A fit of the echo decay data in Fig. 2 produced a T_2 for the coal sample of 211±12 ns. However, it is known that instantaneous diffusion can have an impact on the ESE measurement [7]. Further ESE experiments, where the second pulse width was varied, were performed to check for an effect from instantaneous diffusion. The plot of T_2 as a function of $sin^2(\theta/2)$, where θ is the spin turn angle of the second pulse is shown in Fig.3.

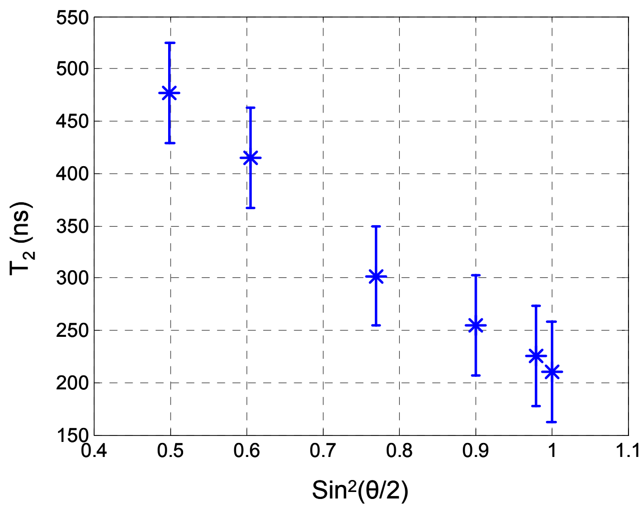


Fig. 3. The T_2 versus $\sin^2(\theta/2)$ of the coal sample.

The linear trend of the plot in Fig.3 indicates that instantaneous diffusion has a significant impact on the sample T_2 measurement. When extrapolated to $\theta = 0$ degrees the linear plot suggests an estimate of the T_2 value, in the instantaneous-diffusion free situation, of 800±80 ns. This T_2 result for the "South Blackwater" coal is within the range 356 ns to 1450 ns seen previously for various coal samples [1].

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