Micro-Coils for MR Spectroscopy by Deep Silicon Etching

R R A Syms¹, M M Ahmad¹, I R Young¹, D Gilderdale¹, D J Collins² and M O Leach²

¹Optical & Semiconductor Device Group, EEE Dept., Imperial College, London SW7 2AZ, UK
²Cancer Research UK Clinical Magnetic Resonance Research Group, Institute of Cancer Research & Royal Marsden NHS Trust, Downs Road, Sutton, Surrey SM2 5PT, UK

Email: r.syms@ic.ac.uk

Abstract. A process for batch fabrication of low-cost microcoils for magnetic resonance spectroscopy is demonstrated. Conductors are fabricated on oxidised silicon substrates by electroplating metals inside a deep photore sist mould, and passivated using an epoxy-based resist. Through-wafer deep reactive ion etching is used to define sample volumes and stencil cuts around each die, and dies are separated by snap-out. Single-coil and multiple-coil sensors are constructed by stacking parts on baseplates fabricated on the same wafer. Single-turn coils have a Q-factor of ≈ 15 at 63.6 MHz, and Helmholtz coils a Q-factor of ≈ 13. Magnetic resonance imaging and spectroscopy are performed, and a SNR of 900 is achieved.

1. Introduction

Recent work has suggested that magnetic resonance spectroscopy (MRS) may be useful in evaluating cancerous lesions [1]. MRS data have been obtained from a variety of body organs including the brain [2, 3]. In a brain tumour resection, many (> 12) biopsy samples may be taken. Clinicians use samples not just to determine the etiology of the tumour, but to define its boundaries. Samples are often cylinders, around 1 mm in diameter and 5 - 10 mm long. The components of interest in MRS (which almost invariably uses protons as the target nuclei) exist in tissue in 0.5 to 5 mmol quantities. To obtain data quickly, close coupling between the sample and the MR detector is required; this implies small coils, which are expensive and require careful handling. One major potential problem is sterilisation between patients, and the ideal solution is a cheap, single-use coil.

The key goal is to maximise the signal-to-noise ratio, so that the advantages obtained from proximity to the signal source are not lost [4]. Coil resistivity and capacitive leakage must be minimised, so that a resonant detector with a high Q-factor may be constructed. The most common frequency in clinical MRI is 63.6 MHz - the resonance frequency of protons in a 1.5 T magnetic field. At 63.6 MHz, the skin depth $\delta = \frac{1}{\sqrt{\pi f \mu_0 / \rho}}$ is around 8.3 $\mu$m for Cu. Conductors around $2\delta = 16 \mu$m thick are therefore used to minimise resistance. Methods of combining thick conductors with substrates that can be etched to provide sample location features are therefore desirable.
Planar MR detectors were first fabricated by surface patterning some years ago [5, 6]. More recently, coils have been combined with sample volumes on glass [7] and deposited on plastic [8], and implantable devices have been developed [9]. The best performance is obtained on insulating substrates [10]. However, modest performance can be obtained on intrinsic silicon, allowing low-cost structuring by deep reactive ion etching (DRIE), a method of near vertical etching which operates by a cyclic repetition of etching and passivation processes [11]. DRIE has a high rate (> 2 μm/min), and may etch through a 500 μm thick wafer in under 4 hours. In addition to forming sample volumes, DRIE may also be used to segment a wafer, thus eliminating the dicing step, which is often dirty, and time-consuming.

In this paper, we show for the first time that deep etching of silicon may be used for the low-cost fabrication of precisely dimensioned MR detectors, based on electroplated conductors surrounding well-defined sample volumes. Section 2 describes fabrication; Section 3 presents the results of electrical characterization and MR experiments with model samples, and Section 4 contains conclusions.

2. Wafer-Scale Micro-coil Fabrication

In our process, micro-coils are formed as shown in Figure 1a. Here a wafer carrying an electroplated conductor (which may either be single-turn as described here, or multi-turn using additional levels of lithography) is etched to define firstly a sample trough and secondly a stencil cut that separates the coil from the wafer, apart from short sections of connecting sprue. The coil is released by fracturing the sprue, avoiding the need for dicing. Other components (e.g., base-plates carrying additional circuitry, as shown in Figure 1b) may be fabricated in a similar way. Figure 1c shows how single coils may be mounted on a baseplate to form a sample trough with a closed base. Figure 1d shows how parts may be stacked to form a Helmholtz coil. One coil component is flip-chip bonded to a baseplate, while a second is stacked on top of the first and wirebonded to complete the coil winding.

![Figure 1. a) Micro coil; b) baseplate; c) coil assembly; d) Helmholtz coil assembly.](image)

Figure 2 shows the fabrication process. A 100 mm diameter <100> oriented, intrinsic (> 5000 Ω cm) wafer is first thermally oxidized to form a 1.5 μm thick insulation layer (step 1). The front side of the wafer is then coated with an adhesion layer of 300 Å Ti and a seed layer of 2000 Å Cu by RF sputtering (step 2). A mould for electroplating is then formed by spin coating a 22 μm thick layer of AZ6290 photoresist (Clariant UK Ltd.). The resist is patterned by UV lithography using the conductor mask, and conductors are formed by depositing a 14 μm thick Cu layer using an acid
plating solution (FB, from Technic Inc.) and a 4 µm thick Au layer using a thiosulphate/sulphite solution (ECF 60, from Metalor) (step 3). The mould is then stripped. A mask for deep reactive ion etching is then formed from a 20 µm thick layer of SU-8 2050 (Chestech, UK), a rugged, epoxy-based resist [12] that also passivates the coil (step 4). Contact pads are protected with a layer of AZ9260. Exposed oxide is then removed using a Plasma Technology System 80’ parallel plate reactive ion etcher, using CHF₃, O₂ and Ar gases (step 5). Deep reactive ion etching is performed using a Surface Technology Systems Single Chamber Multiplex inductively coupled plasma etcher, using SF₆ and O₂ gas for etching and C₄F₈ for passivation. Etching is carried out at a rate of 2 µm/min, until the oxide layer at the rear of the wafer is reached (step 6). The remaining rear oxide is then removed by reactive ion etching in the System 80’. The resist pads covering the contacts is then removed using heated acetone, followed by oxygen plasma cleaning.

![Figure 2. Process for fabrication of micro-coils by deep etching of silicon.](image)

Coils were fabricated with 100 µm wide conductors surrounding 1 mm x 5 mm sample troughs, in an overall die size of 3 mm x 11 mm. Figure 3a shows a completed wafer carrying both coil and baseplate dies. After snap-out, coils were either mounted directly on small PCBs, or were stacked to form Helmholtz coils as previously shown in Figure 1d. Dies were aligned by their edges, and bonded together with epoxy resin. Electrical connections were formed using indium metal as solder. Figure 3b shows (left to right) i) a coil, ii) a coil mounted on a small PCB, iii) a baseplate, iv) a coil attached to a baseplate, v) a Helmholtz coil, and vi) a Helmholtz coil mounted on a PCB.

![Figure 3. a) Completed wafer and b) coil components at various stages of assembly.](image)
3. Experimental results

Electrical performance was measured with an Agilent E5061A vector network analyser. Single coils had an inductance of 9 nH, which rose to 11 nH after mounting on a silicon baseplate. Helmholtz coils had an inductance of 24 nH after wirebonding to a small PCB. Coil assemblies were configured as parallel resonators using surface mount capacitors. High Q-factors were obtained, showing that good performance can still be achieved despite the use of a silicon substrate and soldered interconnects.

Figure 4a shows the frequency variation of the impedance of resonant detectors based on a single-turn coil, configured for operation at 63.6 MHz (i.e., in a 1.5 T field) and 127.2 MHz (3 T). In the former case, Q-factor is 15 and the shunt impedance at resonance is 60 Ω. In the latter case, the Q-factor has risen to 22. Figure 4b shows the frequency variation of the Q-factor, which peaks at ≈25 at around 200 MHz, suggesting that good performance would still be obtained in a 6 T field. Resonant Helmholtz detectors had a Q-factor of 13 when configured for operation at 63.6 MHz.

MR experiments were performed using a 1.5 T Siemens Magnetom Vision MRI system. The Vision system body coil was used for RF transmission and the microcoils for signal reception. The microcoils and test samples were placed inside a large body coil loading annulus. Microcoils are sensitive to both interior and exterior signal sources, and interior imaging was first demonstrated with a source of mobile protons in the sample volume. The material used was taken from a “Spenco” dermal pad, a vinyl plastisol material from Spenco Healthcare International, Horsham, West Sussex, UK which has proved to be an effective signal source for use in coils designed to provide marker signals in MRI.

Exterior MR imaging was then demonstrated. Figure 5a shows a coronal slice image of a Spenco sample obtained using a single-turn coil. The slice is a horizontal cut through a sliver of Spenco measuring 3 x 4.5 x 0.7 mm, standing vertically within the sample volume. The images were based on a 28 x 28 mm field of view, a 500 µm slice thickness and four signal averages from a 35 ms spin echo sequence. Excellent SNR is clearly obtained. Figure 5b shows a similar image obtained from a Helmholtz coil.

Exterior MR imaging was then demonstrated. Figure 5c shows a transverse slice image of a semi-cylindrical capsule of cod liver oil, placed directly above the single turn coil. The capsule diameter was approximately 8 mm. The majority of the capsule perimeter may be seen, but the signal falls off above the plane of the coil, falling to around 10% of its peak at around 2.4 mm above the coil. This
image was obtained using a 56 x 56 mm field of view, a 500 µm slice thickness and two signal averages of a 35 ms spin echo sequence.

**Figure 5.** Arrangements and MR images from samples of a), b) Spenco and c) cod liver oil.

Magnetic resonance spectroscopy of both types of sample was then demonstrated. Figure 6a shows a MR spectrum obtained from the Spenco sample using a single turn coil. The spectrum consists of a large, narrow peak with two clearly resolved side peaks, which is characteristic of this material. The measured linewidth of the main peak was 54 Hz. Figure 6b shows a similar spectrum for the cod-liver oil sample. The linewidth of the lipid peak is 12 - 17 Hz, and the SNR (estimated from the peak signal divided by the RMS value of the noise in the absence of signal) is ~ 900. The data shown here have been apodised to boost high frequencies; this procedure helps to identify doublets, which can otherwise appear as a single broad peak. Considerable structure may be seen in the oil spectrum, and preliminary assignment of the different peaks is in progress.

**Figure 6.** MR spectra obtained from samples of a) Spenco and b) cod-liver oil.

4. Conclusions
We have demonstrated a wafer-scale process for batch fabrication of magnetic resonance detectors on oxidised silicon substrates. The conductors are formed first, by electroplating metals inside a photoresist mould, and the die topography is then defined by deep reactive ion etching. Detectors
may be detached from the wafer by a simple snap-out process and assembled into a variety of configurations, including single turn and Helmholtz coils. Good electrical performance has been demonstrated, with Q-factors up to 15, despite the use of a semiconducting substrate. Preliminary MRI and MRS experiments have been performed, and excellent SNR and linewidth have been obtained. This level of performance is extremely encouraging, and confirms the essential validity of the fabrication scheme. However, further improvements in signal obtained from optimised multi-turn coil geometries and from an increase in magnetic field strength (and hence in operating frequency) are expected. An obvious near-term target is to perform similar evaluations of tissue samples, and this work is in progress. To avoid difficulties with sample insertion, additional needle coil geometries are also being investigated.

Acknowledgements
The Authors are extremely grateful to EPSRC for funding under grant GR/S08077/01 and also to Dr John Stagg for performing the deep reactive ion etching used in this work.

References