Needle Micro-Coils for MRS

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Abstract - A process for batch fabrication of low-cost needle-shaped microcoils for magnetic resonance (MR) spectroscopy is demonstrated. The conductors are embedded inside a cross-section designed to avoid the signal cancellation effects that can occur with a completely immersed coil. Conductors are fabricated on oxidised silicon substrates by electroplating metals inside a deep photoresist mould, and then capped with a thick layer of epoxy-based resist. Through-wafer deep reactive ion etching is used to define needle shapes, and die separation is carried out by snap-out. Prototype coils are mechanically robust, and sharp enough to puncture model samples of fruit. At 63.8 MHz, Q-factors obtained on Si substrates are comparable to those obtained on glass, and resonators based on single-turn coils have Q-factors of 14. Total immersion 1H MR imaging and spectroscopy are both demonstrated in a 1.5 T field using tomato fruits.

I. INTRODUCTION

There are very few instances in human magnetic resonance (MR) studies in which the RF detector is immersed in the tissue to be examined, and virtually none in which there is no effective spatial localisation. Most examples arise with small coils for catheter localisation [1]. Generally the spatial encoding process means that spins in individual voxels are uniquely encoded so that they may be differentiated, and the signals detected by the immersed coil reflect this process.

If a small coil is completely immersed in a tissue containing uniformly distributed metabolites (Figure 1a), the signals from some locations can be almost entirely cancelled by those from the same metabolite in other regions. Since the field of view of small coils is limited, and their sensitive volume is small, the spatial encoding used for magnetic resonance spectroscopy studies in vivo is normally irrelevant. Metabolites used in most spectroscopy studies are present in tissue in millimolar concentrations. Thus, the voxel sizes needed are much larger than the sensitive volume of the coil. This problem can be addressed by moving the detector very close to the target nuclei. The “filling factor”, which reflects the tightness of the coupling between nuclei and detector, appears as a linearly proportional factor in the signal-to-noise ratio relationship.

Here we describe a needle-shaped coil designed for direct insertion into tissue. Though the sensitive volume of such a coil is so small its design must be such that it can be used for MR spectroscopy as well as imaging. Such a coil could find a valuable role in the resection of (for example) brain tumors under MR guidance. Here the surgeon is seeking to confirm whether all the malignant tissue has been removed, and is using the differing distributions of metabolites between normal and malignant tissues as markers [2]. Low-cost fabrication is required so the surgeon may test many sites round the resected tumor cavity, using each needle only once before disposal.

Planar MR detectors were first fabricated on GaAs and glass some years ago [3]. Surface coils have been combined with microfluidic analysis such as capillary electrophoresis [4]. Considerable efforts have been devoted to improving coil plating processes [5], and planar coils have been formed on flexible substrates and integrated with capacitors [6]. The best performance is obtained on insulating substrates [7]. However, usable performance can also be obtained on silicon, which potentially allows co-integration of the components such as diode switches and amplifiers. In practise, Q-factors are limited by capacitative coupling through the lossy substrate, which gives rise to an additional lossy parasitic resonance. These problems are well understood in RF IC design [8], and have been addressed by several methods including undercutting.

The use of Si also allows low-cost structuring by deep reactive ion etching (DRIE), a method of near vertical etching that operates by a cyclic repetition of etching and passivation [9]. Coils may be passivated with SU-8, an epoxy-based resist [10] with excellent mechanical and dielectric properties. Recently, we have shown that coils
formed this way may have sufficient performance for both imaging and spectroscopy [11]. Here, we show how the approach may be used to form needle-shaped coils. We base the design on the reciprocity theorem [12], and use this to identify needle cross-sections that avoid signal cancellation. Section II presents a simple theory for total immersion coils. Section III describes fabrication, characterisation and 1H MR imaging and spectroscopy experiments using model samples. Section IV contains conclusions.

II. THEORETICAL ANALYSIS

First, we develop an elementary theory to estimate the sensitivity of a needle-shaped receiver, which is assumed to be a long, thin rectangular coil mounted on the centreline of a passive support with a rectangular cross-section, as shown in Figure 1b. The coil is sufficiently long that its ends may be neglected, and the analysis is based entirely on the conductors forming the long sides, which are assumed to be cylindrical wires of radius r. For simplicity we only consider a single turn. The conductors are 2a apart and the support measures 2w x 2h. The task is to choose w and h to maximise the detected signal obtained when the coil is surrounded by a signal source consisting of a uniform distribution of precessing nuclear dipoles.

We assume the coil intercepts the y-component of the signal field. From reciprocity, we assume that the signal induced by a uniform dipole distribution occupying a given area is proportional to the integral of the y-component H_y of the magnetic field H that would be created by the coil over the same area in transmit mode. If the integration is over all space, the result is zero for each conductor in isolation, and for both conductors together. If the coil is completely immersed in a uniform dipole distribution, the signal received must therefore also be zero. However, a non-zero signal may be obtained by excluding dipoles from particular regions using a shaped coil support. In low magnetic fields, when the frequency is low, we may identify a suitable cross-section using static analysis. If the coil carries a current I, H_y may be found using Ampere’s law for x^2 + y^2 > r^2 as:

\[ 2\pi I_h / I = (x + a) / ((x + a)^2 + y^2) - (x - a) / ((x - a)^2 + y^2) \]

(1)

Figure 1c shows contours of equal H_y obtained from Equation 1. The map is divided into areas for which H_y is positive (shaded grey) and negative (white). The dividing contour is that for which H_y = 0, namely the two hyperbolae x^2 - y^2 = a^2. Since the integral of H_y over all space must be zero, the integrals of H_y over the grey area A and the white areas A’ and A” together must be equal in magnitude but opposite in sign, so they cancel on addition. Maximum signal must then be obtained in two cases, when the source is localised within A (which corresponds to a coil sensing an internal sample) or within A’ and A” (an external sample). The optimum cross-sectional shape for the coil support is therefore the area A itself. This shape is clearly impractical.

The principle of complementarity implies that the same magnitude of signal will be obtained in geometries such as Figure 2a (where the source occupies a given area AS inside the coil) and Figure 2b (where the source is now excluded from the same area AC). The signs of the detected signals will be opposite, however. In general, the sensitivity S relative to the optimum may therefore be calculated by comparing the integral of H_y over the region AS in Figure 1c with the integral over A. Thus, we may write:

\[ S = I_{AS} / I_A = - I_{AC} / I_A \]

(2)

Here the integral over a region R is defined as I_R = \int_R H_y \, dx \, dy. We have carried out the integration needed to evaluate Equation 2 numerically. To do so, we limited the regions A and AS in the x-direction to lie inside the conductors, and took the limit of a small conductor radius r. The integral I_A does not converge, rising logarithmically as the extent of A increases in the y-direction. Since the total integration region will be limited for purely practical reasons, such as the size of the sample or the sampling volume, we arbitrarily limited the y-range to |y/a| ≤ 10.

Figure 2c shows the variation of |S| with h/a, calculated assuming that w/a = 1. The sensitivity rises monotonically from zero as h/a increases. The limiting value (obtained when the coil is effectively mounted in a vertical slab of width 2a) is around 0.77. Thus, an infinite slab, set upright, will capture about 3/4 of the signal from the surrounding sources. When h/a = 1 (when the coil is mounted in a square cross-section) around 0.43 of the signal is obtained. Thus, a square section will capture much of the signal available. This geometry is easily achievable, and might correspond (for example) to a pair of conductors on a 1 mm spacing with a 0.5 mm thick substrate and a 0.5 mm thick cap layer.

Clearly, the omission of the upper half of the coil support will halve the detected signal. A lower signal will also be obtained if the coil support width is reduced so that w < a. A reasonable assumption might be that increased signal would be obtained when w > a. However, we have found that the improvement obtained by blocking more of the grey area in Figure 1c is outweighed by the rapid reduction in signal from the white area, since the sensitive regions near the conductors now detect less. It is certainly possible to refine the design to capture more of the available signal, for example by replacing the simple square cross-section using an “I-section” that mimics more closely the grey area in Figure 1c. However, the increased needle size could make for difficult insertion. A simple square section with w = a is therefore close to optimum.
III. FABRICATION AND TESTING

Prototypes based on square sections similar to Figure 1b were formed from a coil mounted on a silicon substrate and capped with plastic. Although the plastic itself contains protons, these will not generate a significant signal without using the specialised techniques of solid-state MR. Needle coils were formed with the cross-section shown in Figure 3a. The conductor width and height were 100 μm and 18 μm respectively. The centre-to-centre conductor separation was 2a = 1 mm, while the overall needle width was 2w = 1.3 mm, so that the overall cross-section was close a square. The substrate thickness was TS = 550 μm and the thickness of the SU-8 cap was TC = 500 μm, so that h = TS = TC. Layouts were as shown in Figure 3b. Each die consisted of a coil surrounded by a trepan cut, so that the resulting devices were needle-shaped in one plane and rectangular in the other. The cut was interrupted to form a short ‘sprue’ that attached the coil to the wafer during processing. Four device variants were formed: single- and double turn coils, and short and long coils. Double-turn coils were formed using single-layer metallisation and a wire-bonded bridge. Short coils had shaft lengths L1 = 5 mm and tip lengths L2 = 2.5 mm; short coils had L1 =10 mm and L2 = 2.5 mm.

Prototype were formed using the two-mask process in Figure 3c. A 100 mm diameter <100> oriented, intrinsic (~5000 Ω cm) wafer was first thermally oxidised to form a 1.5 μm thick SiO2 layer (step 1). The front side of the wafer was then coated with an adhesion/seed layer of 300 Å Ti and 2000 Å Cu by RF sputtering (step 2). A mould for electroplating was then formed by spin coating a 22 μm thick layer of AZ6290 photoresist. The resist was patterned using the conductor mask, developed in AZ400K developer, and hard baked (step 3). The exposed seed layer was cleaned using an acid dip. Standard formulae were used to estimate a suitable conductor thickness. The skin depth is δ = 1/(√(f0μ0ρ)), where ρ is the resistivity of the conductor material, f is the frequency and μ0 = 4π x 10-7. The operating frequency f0 for H MR scales linearly with magnetic field at 42.57 MHz/T. In a 1.5 T system, f0 = 63.8 MHz. For Cu, we then obtain δ = 8.3 μm, suggesting that a thickness of ~16 μm will be suitable. Conductors were formed by depositing a 14 μm thick Cu layer using an acid plating solution and a 4 μm thick Au layer using a thiosulphate/sulphite solution (step 4). The mould was then stripped, and exposed seed metal removed by sputter etching (step 5).

A mask for deep reactive ion etching was then formed. A 500 μm thick layer of SU-8 2050 was first deposited by spin coating over the conductors (step 6). The SU-8 was patterned using the structural-layer mask, developed, and hard baked (step 7). Exposed areas of oxide were then removed using an parallel plate reactive ion etcher. The trepan cuts were formed by deep reactive ion etching, with a STS inductively coupled plasma etcher. Etching was carried out at a rate of 2 μm/min, until the oxide layer at the rear of the wafer was reached (step 8). The remaining rear oxide was then removed (step 9). Figure 4a shows the four coil variants. Here, the coils may be seen through the optically transparent SU-8 capping layer. Figure 4a shows a scanning electron microscope view of a long single-turn coil.

Mechanical strength was established by repetitively inserting a needle coil into various fruits. No significant damage was observed, even after 50 insertions into a hard green apple (Figure 5a). Durability was assessed using several cleaning schedules that might be used in sterilisation. 1. Components were immersed in different solvents (acetone, methanol, water, ethanol, isopropanol, trichloroethane and chloroform) for 5 days, and then ultrasonically cleaned at 50W power, 50-60 MHz frequency for 10 minutes. No change was observed for the last five solvents, but the SU-8 layer peeled off after immersion in acetone and methanol. 2. Components were exposed to steam at for 60 min, and immersed in boiling water for 30 min. No change was observed in the former case, but the SU-8 layer delaminated in the latter. 3. Components were heated on a hot plate for 10 min, and cooled suddenly to room temperature. No change was observed for temperatures up to 100°C; however, the SU-8 epoxy changed colour at 150°C, and delaminated at 200°C. 4. Components were exposed to UV (405 nm) radiation at 4 mW/cm2 for 10 min; no change was observed. 5. Components were bombarded by O2 plasma at 200 W RF power, 60 scm flow rate and 50 mTorr pressure in an parallel plate RIE for 10 min. No change was observed. These results suggest that the coils are extremely durable.

Figure 4. a) optical and b) SEM view of needle coils.

Figure 5. Needle coil puncturing a) apple and b) tomato.
Electrical performance was evaluated using an Agilent E5061A network analyser. The inductance of long single-turn coils was 19.3 nH, rising to 27.5 nH after wirebonding. At low frequencies, the Q factor rose quasi-linearly. However, above $\approx 50$ MHz the rate of increase slowed due to substrate losses and the Q-factor peaked at $\approx 100$ MHz. At 63.8 MHz, the use of a Si substrate caused little Q-factor penalty. L-C resonators were constructed for operation at this frequency using non-magnetic capacitors. For long single-turn coils the Q-factor and impedance were 14 and 120 $\Omega$, respectively. Two-turn coils had higher inductances, but their Q-factors were not significantly higher than single-turn devices. For magnetic resonance experiments, coils were attached to small printed circuit boards. Coils were matched down to 50 $\Omega$ impedance using an additional capacitor, and a PIN diode (which had a DC bias applied through the same pair of conductors) was used with an additional inductor to detune the coil during RF excitation.

$^1$H magnetic resonance experiments were performed using a 1.5 T Siemens Magnetom Vision MRI system. The main body coil was used for RF transmission and the microcoils were used for signal reception. The microcoils and test samples were placed inside a large body coil loading annulus. A model sample was provided by a baby plum tomato, into which a long needle coil was inserted as shown in Figure 5b. MR imaging was demonstrated first. MR signals were obtained from the coronal plane, using a spin echo sequence with a 500 $\mu$m thick slice and a 56 mm x 56 mm field of view and a 256 x 256 imaging matrix. The repetition and echo time $T_R$ and $T_E$ were 600 ms and 35 ms, respectively. Four signal averages were used. Figure 6a shows an axial image of the tomato impaled on the needle coil. The opaque centre is the coil position, and the bright surrounding patch is signal obtained from the tomato. This figure demonstrates that the coil is capable of receiving a non-zero signal even when totally immersed in a signal source. The lateral range of the signal is approximately equal to the conductor separation (1 mm).

MR spectroscopy was then demonstrated. MR signals were again acquired from the coronal plane, this time using a localized point resolved spectroscopy (PRESS) sequence based on slice-selective 90° RF excitation, dual slice-selective 180° RF refocusing and acquisition of the resulting tertiary spin-echo. Water suppression was achieved using a single 60 s chemically selective (H$_2$O) Gaussian radio frequency pulse followed by gradient spoiling. The sample volume was 15 mm x 15 mm x 15 mm with an overall field of view of 56 mm x 56 mm. The repetition time was $T_R = 1500$ ms, the echo time was $T_E = 144$ ms, and 128 sample averages were used. Figure 6b shows a spectrum of baby plum tomato in the region between 4.5 ppm and 2.5 ppm $^1$H shift. The spectrum shows various fructose and glucose groups is consistent with recent high field measurements of tomato fruits. The SNR (the ratio of the peak signal to the root mean square noise) is $\approx 40$. The H$_2$O linewidth is an extremely encouraging 12 Hz.

![Figure 6. a) MR image and b) MR spectrum of tomato.](image)

### IV. Conclusions

This result demonstrates that the prototype coils can obtain viable quantitative MR data. Further development is clearly needed to improve Q-factors at the higher frequencies of high-field MRS.

### REFERENCES


