Microcoils for NMR microprobes with improved SNR

Miniaturisation of NMR probes has been the latest trend which allows portability and analysis of the chemical composition of minute (nL-µL) volumes of sample. NMR experiments typically require that such probes are used in external setups with very strong DC magnetic field and external detector circuits. A critical aspect for the success of such an experiment is to ensure a high signal-to-noise ratio (SNR). However, the resulting value of the SNR delivered by a miniature probe has a complicated dependence on both the external measurement setup, chemical composition of the sample and the probe design. For probe designers there are no clear guidelines to suggest how a given probe will behave under any external measurement setup or chemical composition of the sample under test, or which design factors can be modified to optimize the probe's SNR.

In this thesis, we first begin with formulating a clear guideline on how to design such a microprobe from an electronic engineering perspective. We considered that both the radiofrequency (RF) magnetic field generator and detector is a planar microcoil embedded in silicon with a sample holder on top of it. Detailed electromagnetic simulations of microcoil structures and sample holders revealed the effects of different parameters that may affect the SNR. This analysis led us to the introduction/definition of a totally original and novel parameter: the performance parameter P, whose maximization (by changing a microprobe's design) optimizes the resultant SNR in *any* external setup. Several microcoils of 'large' and 'small' dimensions (depending on the size of the overall surface area covered by the respective microcoils) as well as of different aspect ratios of the conductor have been simulated in detail and the performance factor P was calculated in each case.

Next, these coils were fabricated in a high resistivity silicon wafer through electrodeposition of copper inside DRIE trenches initially etched into the substrate. However, post-DRIE scallops posed a severe problem for the continuous deposition of the Cu seed layer in the subsequent step. Hence, an inventive trench wall planarization process has been developed that significantly reduced (with 80%!) the roughness of trench walls, enabling the subsequent seed layer deposition without any problems.

The copper electroplating process also had to be optimized using an orthogonal design of experiments (DOE) which determined the deposition parameters necessary to fill the realized trenches without voids. Different optimizations had to be carried out for copper filling of small (trench 7 µm wide) and large (trench 25 µm wide) trenches as initial I-V measurements revealed a very high resistance that was ultimately found to had been caused by voids inside the filled trenches. For these first batch of coils a novel wet etching technique using sodium persulphate was developed, optimized using another set of orthogonal DOE, to remove the excess Cu overburden post CMP. However, although this method resulted in a fast etch rate, it also exhibited high etching non-uniformity. Moreover, when used on actual device wafers it also unpredictably and non-uniformly etched the Cu inside the trenches wherever the etchant was able to seep in through the cracks and voids of the copper layer

For the second batch of coils which consisted of different aspect ratios viz 1:3, 1:1 and 3:1 progressive electroplating followed by CMP (for which recipe was again developed) was used for excess copper removal. However, finally a combined CMP + short persulfate dip process was adopted to ensure good planarization and removal of excess copper.

These coils were tested under probe station for IV characteristics as well as under low frequency Q values were measured. Furthermore, magnetic field from these coils were also measured and compared with the simulated results.