3D MICROCOIL FABRICATED ON THE CAPILLARY SURFACE BY CYLINDRICAL PROJECTION LITHOGRAPHY FOR NMR APPLICATION

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ABSTRACT

In the present work, a 3D gold microcoil has been directly fabricated on a capillary surface based on MEMS technology utilizing our new developed cylindrical projection lithography system. Compared with other hand winding, laser exposure and contact printing 3D microcoils, the one our reported can realize higher fabrication resolution and efficiency. As a result, this microcoil would greatly improve sensitivity of nuclear magnetic resonance (NMR) used in detecting mass-limited samples.

KEYWORDS

NMR, 3D microcoil, MEMS, Cylindrical projection lithography

INTRODUCTION

Nuclear magnetic resonance (NMR) is a powerful analytical tool, known for its ability to identify chemical and biological signatures. However, its relative lack of sensitivity has limited the use of NMR for studies of mass-limited samples. Numerous strategies over the years have been investigated to increase sensitivity of the NMR experimental, including winding small copper wire on the thin capillary [1], inkjet contact printing and laser exposure to form 3D microcoils, etc. Recently, even several planar microcoils have been developed using microelectronic and micromachining techniques [2]. However, above-mentioned microcoils have lower fabrication resolution and magnetic field inhomogeneity problems, respectively. With regards to this, here we developed a new fabrication method of 3D microcoil on the thin capillary based on our previous cylindrical projection lithography system [3]. The magnetic field strength distribution of the 3D coil on capillary has been simulated and analyzed using Ansoft Maxwell commercial software. The main fabrication processes, such as spray coating, magnetron sputtering, wet etching and cylindrical projection lithography were used to realize the microcoil on capillary. Finally, the fabricated microcoil was characterized by a network analyzer and its S_{11} reflection coefficient was also tested.

DESIGN AND SIMULATION

The layout and circuit using microcoil for on-line NMR detectors is shown in figure 1(a), C_t and C_m are the tuning and matching capacitors, respectively. The microcoil is used as a key radio frequency (RF) component in NMR probe. Maximum sensitivity for the solenoidal microcoil is achieved by keeping the coil axis vertical to the applied magnetic field. A corresponding RF circuitry mainly consists of capacitors is connected with two electrodes of the microcoils. Tested mobile phase or buffer fluid can flow in the capillary with microcoil. The designed microsystem can be used to detect and analyze mass-limited samples, even nanoliter-volume ones for portable and low-cost NMR equipment applications. Figure 1(b) shows the section view of the capillary with microcoil. The outside diameter (OD) of capillary is 1mm and the inside diameter (ID) is 800μm. In the present work, the coil will be fabricated by sputtering gold (Au) material and subsequent wet etching process. The thickness of microcoil is 0.15μm. The main structural parameters of the capillary and designed microcoil are indicated in figure 1(c). The number of turns of whole coil structure is 20, and its line width and spacing are both 30μm.

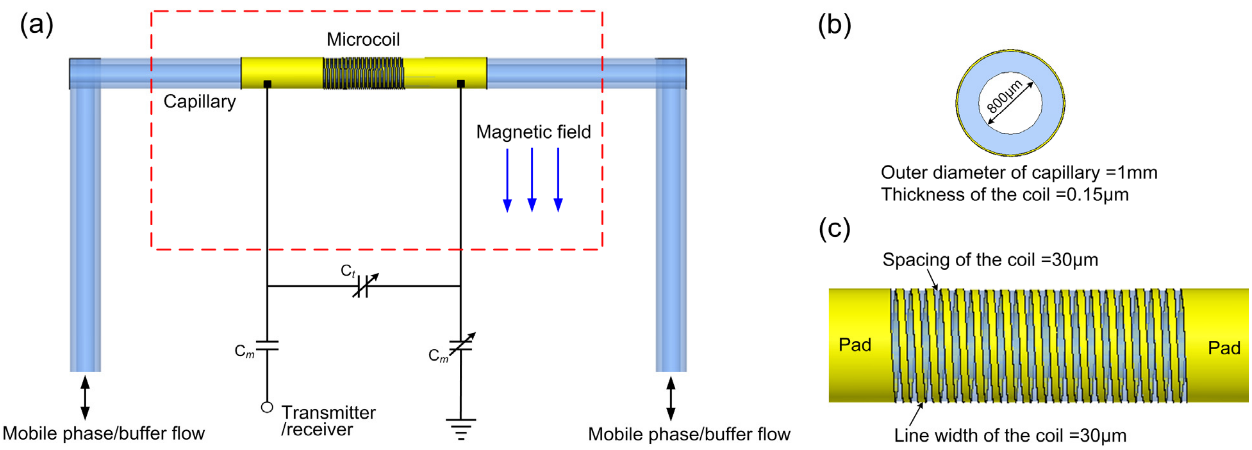

Figure. 1 Schematic diagram of (a) layout and circuit using microcoil for NMR detection; (b) section view and (c) main structural parameters of the designed microcoil on a capillary surface

The magnetic field strength distribution of the microcoil on capillary is simulated by Ansoft Maxwell commercial software for analyzing its distribution uniformity. Figure 2 shows the simulated magnetic field strength distribution of the 3D microcoil under certain 1A direct current. The maximum magnetic field strength is about 0.024T. It can be seen that the magnetic field around the capillary shows good homogeneity, which is important for realizing a stable and reliable detection.

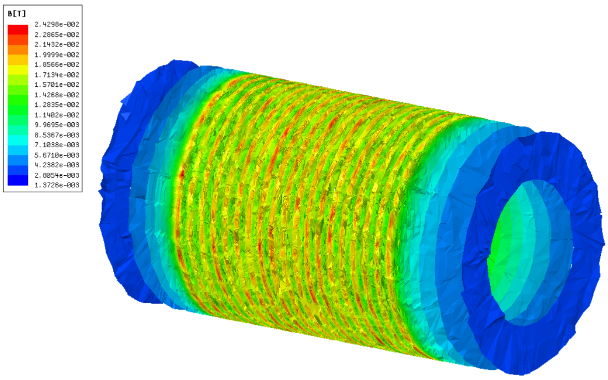

Figure. 2 Simulated magnetic field strength distribution of the 3D microcoil on capillary

MICROFABRICATION AND CHARACTERIZATION

Figure 3 is the basic fabrication process of the microcoil. After Au film sputtering the spray coating resist film was baked at 120°C for 20min. The cylindrical projection lithography was done for the patterning the resist film utilizing our previously developed cylindrical projection lithography system [3]. Then the Au film wet etching process was carried out successively, and main procedures are as follows:

(a) Capillary substrate ready and cleaning the surface of the capillary using plasma;
(b) Au film with about 0.15μm thickness was sputtering onto the capillary surface by magnetron sputtering;
(c) Spray coating resist on the capillary substrate by developed home-made coating system with heater nozzle [4];
(d) Exposure and patterning of the capillary with coated resist film by developed cylindrical lithography method;
(e) Au film coated on the capillary was patterned by wet etching process;
(f) Resist film was removed in acetone and obtaining the solenoidal microcoil on the capillary.

In above-mentioned microfabrication processes, the used photoresist is S1830 (Shipley Co. LLC) which is one of the most commonly used resists in spin coating process at our laboratory. In addition, AZ5200 (AZ Electronics Materials) is used as a thinner solvent. This thinner is mainly based on propylene glycol monomethyl ether acetate (PGMEA), which is commonly utilized as a thinner solvent for the direct spray coating process. All resist solutions were prepared in weight ratio just before the spray coating process.

The quartz capillary substrates used in this work were all cleaned by immersing in the H_2SiO_4/H_2O_2 solution at 115˚C for 15 min and then rinsed with purified water. Then they were treated by a UV ozone treatment unit (AcingTec. VX-0200HK-002) for the surface modification. The cleaned cylindrical substrates were loaded into the spray coater and spray deposited with the prepared resist solutions. Systematic experiments were carried out aiming to investigate the effects of real-time heating treatment and rotation speed (0~1500 rpm) of the cylindrical substrate, etc. The film thickness was directly measured by the confocal microscope (Lasertec, OPTELICS H300) and the scanning electron microscope (SEM).

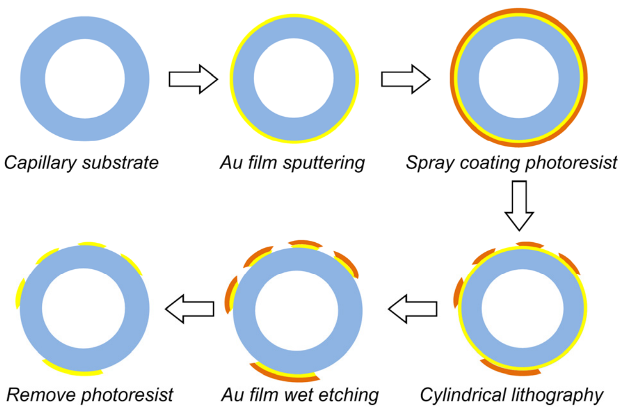

Figure. 3 Main fabrication processes of the microcoil

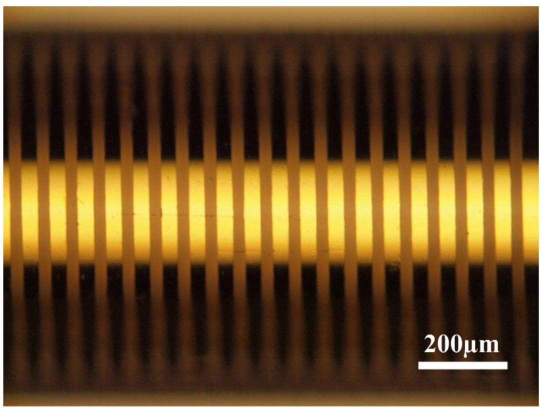

Figure. 4 Optical photo of the fabricated microcoil

In fabrication, the Au film is deposited on the cylindrical capillary surface by two times opposite placement within a chamber where a turbo molecular pump is used to evacuate the chamber to 7.0×10^{-4}Pa. The sputtering gas was argon of 99.9% purity and Au target is 70mm in diameter and of 99.99% purity. The applied power is set to 100W, resulting in a relatively fast growth rate. With these parameters for deposition, we get Au film that is uniformly distributed and very fine.

In cylindrical projection lithography, UV-light strength 1300µW is used to realize the exposure to coated resist with 7µm thickness. In the exposure the rotation speed of the capillary with resist coated is set to 0.2deg/s. After the projection exposure, the development was carried out and residual resist was also removed in acetone. As a result, the resist film was patterned successfully. Then, the Au film without resist coated and corresponding seed layer Cr were removed by wet etching method.

Finally, the successfully fabricated microcoil on the 1mm-in-diameter quartz capillary is shown in figure 4. The measured pitch along longitudinal direction is generally 65µm, while the line width is not constant along the whole range. It is mainly due to the exposure energy is not enough uniform within the exposure area, which is mainly related to the misalignment of the optical axis.

Figure 5 shows the optical photo of the fabricated Au microcoil on a capillary next to a European coin for reference. It can be seen that at two sides of the microcoil there are two pads fabricated for subsequent RF measurement. In the present work, Agilent-4395A network analyzer was used to measure the quality factor of the fabricated microcoil. The quality factor is defined as the resonance frequency divided by its bandwidth, which is about ~8. Figure 6 shows the results of S_{11} refection coefficient measurements. It can be seen that the microcoil is tuned to have a maximum power transfer at 55.6 MHz.

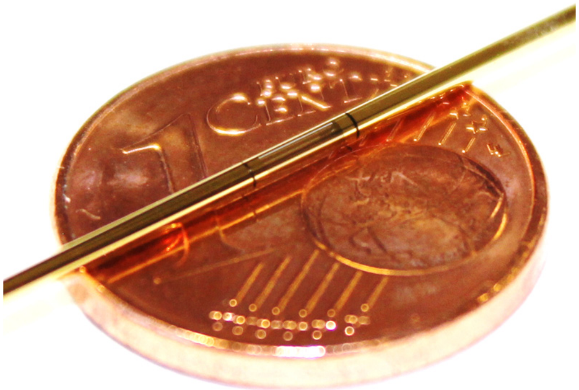

Figure. 5 Optical photo of the fabricated Au microcoil on a capillary next to a coin for reference

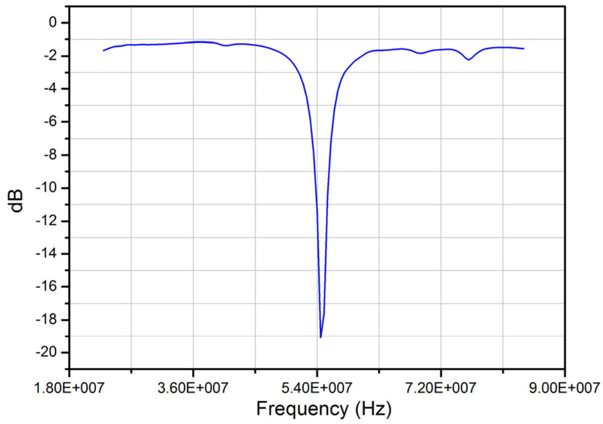

Figure. 6 Test S_{11} reflection coefficient of the fabricated microcoil by network analyzer

CONCLUSION

A new fabrication method of 3D microcoil on a capillary has been proposed. The magnetic field strength distribution of the coil on capillary has been simulated. The basic MEMS fabrication processes were used to realize the microcoil on capillary. The fabricated microcoil has 30µm line width and 60µm pitch. Compared with other traditional methods, the one our reported can realize higher fabrication resolution and efficiency. Finally, the fabricated microcoil was characterized by a network analyzer, which shows the microcoil at 55.6 MHz has a maximum power transfer.

In our further work, the process will be optimized to fabricate higher aspect-ratio microcoil. The detector based on the microcoil will be applied to on-line NMR system with permanent magnetic field for analyzing mass-limited samples.

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REFERENCES

[1] D.L. Olson, T.L. Peck, A.G. Webb, R.L. Magin, and J.V. Sweedler, *High-Resolution Microcoil ^{1}H-NMR for Mass-Limited, Nanoliter-Volume Samples*, Science, pp. 1967-1970. (1995).
[2] C. Massin, F. Vincent, A. Homsy, K. Ehrmann, G. Boero, and R.S. Popovic, *Planar Microcoil-Based Microfluidic NMR Probes*, Journal of Magnetic Resonance, pp. 242-255. (2003).
[3] D. Lee, H. Hiroshima, Y. Zhang, T. Itoh, R. Maeda, *Cylindrical Projection Lithography for Microcoil Structures*, Microelectronic Engineering, pp. 2625-2628. (2011).
[4] Y. Lu, Y. Zhang, J. Lu, A. Mimura, S. Matsumoto and T. Itoh, *Three-Dimensional Photolithography Technology for a Fiber Substrate Using a Microfabricated Exposure Module*, Journal of Micromechanics and Microengineering, pp. 125013. (2010).

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