Environmental Management Science Program

Project ID Number 60247

Miniature Nuclear Magnetic Resonance Spectrometer for In-Situ and In-Process Analysis and Monitoring

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June 1, 1998

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Research Objective

The purpose of this research project is to develop a miniature Nuclear Magnetic Resonance (NMR) spectrometer. This analytical instrument would potentially be used for in-situ, in-field and in-process monitoring, characterization and identification of various chemical compounds. The intended spectral resolution of the NMR spectrometer is better than 0.1 ppm (parts per million). The development of the miniature NMR spectrometer will rely, in part, on the microfabrication technology that includes photolithography, chemical etching, thin film processing and other techniques originally developed in microelectronics. Miniaturization of the NMR spectrometer will involve the miniaturization of the inductive NMR probes used to pick-up the NMR signals. It will also involve integration of the probe, sample holder and magnetic field correction (shimming) coils into a single miniature system. Lastly, it will involve the development of a hand-held permanent magnet capable of creating magnetic field of over 1 Tesla with uniformity of about 1 part per million over the extent of the sample. The advantage of the developed instrument will not only be its portability, but also its ability to handle nono-liter quantities of fluids.

Research Progress and Implications

As of today, the following tasks have been completed.

1) Scaled versions (800 micron diameter) of the NMR inductive probes have been designed, fabricated and tested for their radio frequency properties. The resulting probe design, which can be called a scroll coil, has been found to be suitable for further miniaturization and compatible with the existing microfabrication technology. In addition tests and analysis indicate and this design will also provide higher sensitivity to NMR signals per unit volume of the sample than other proposed miniature NMR probes. Analysis and tests of the scroll coil design revealed a surprising fact that the sensitivity of the scroll coil is much better when the conductor thickness is smaller than the conductor skin depth. This result defies the conventional rules of NMR probe design which prescribe that conductors should be thicker than their own skin depth.
2) As samples and NMR probes become smaller, the problem of the NMR spectrum dispersion caused by the mismatch in the magnetic susceptibility of the sample and its surroundings becomes more pronounced. A method and a computer code for numerical modeling of the NMR spectrum dispersion has been developed. Upper bounds on the errors in the spectrum calculation have been derived. The developed numerical techniques have been applied to design NMR probes that introduce as little distortion as possible to the NMR spectrum. These designs rely on passive shimming (magnetic field correction) by specially patterned parts of the coil.
3) Initial evaluation of a commercially available permanent magnet (from Drexel Corporation) capable of delivering a 2 Tesla field has been carried out using numerical modeling.

Planned Activities

1) Complete miniature NMR probe design fabrication and testing by 12/98. This involves fabricating double layer films consisting of dielectric (polyimide) and conductor (copper).

The challenge is to make conductor thickness between 2.5 and 5 microns uniformly. Another step needed for the completion of the NMR probe is the improvement of the developed rolling technique allowing to reduce the probe diameter down to 100 microns. The final test of the probe should be to obtain NMR spectrum in a conventional spectrometer.

2) Complete evaluation and design of the permanent magnet by 5/99. This involves, most likely, modifying the magnet available from the Drexel Corporation.
3) Evaluate stability of the magnet and design temperature stabilizing system around it by 12/99.
4) Design the magnetic field correction (shimming) coils to be integrated into one system with the NMR probe by 9/99. Fabricate and test these shimming coils together with the NMR probe by 5/2000.
5) Put together the miniature NMR system including NMR probe, sample holder and shimming coil assembly and the permanent magnet with the temperature stabilizing system by 9/2000. Testing of the miniature NMR system should be completed before 12/2000.