

ISSN 1726-5749

SENSORS & TRANSDUCERS

vol. 90
Special
4/08



Modern Sensing Technologies

International Frequency Sensor Association Publishing





Sensors & Transducers

Special Issue
April 2008

www.sensorsportal.com

ISSN 1726-5479

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Special Issue
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www.sensorsportal.com

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A Portable Nuclear Magnetic Resonance Sensor System

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Received: 15 October 2007 / Accepted: 20 February 2008 / Published: 15 April 2008

Abstract: Nuclear Magnetic Resonance (NMR) is a relatively complex technique and normally requires expensive equipment, however with advances in computing, electronics and permanent magnet technologies, NMR is becoming more feasible as a non-invasive tool for industry. The strength of NMR is its ability to probe at the molecular level and hence gain information about molecular structure, organization, abundance and orientation. This paper presents some of the work being undertaken in developing portable NMR systems for the non-destructive testing of materials such as polymer composites, rubber, timber and concrete. *Copyright © 2008 IFSA.*

Keywords: Nuclear magnetic resonance, Non destructive testing, Permanent magnets

1. Introduction

The Nuclear Magnetic Resonance (NMR) is one of the more recent sensing technologies and has become very popular for its ability to non-invasively probe down to the molecular level the properties of many materials and living organisms. Its greatest impact has been in the areas of chemistry and medical radiology, but now it is being applied to biochemistry, structural biology, and materials science research [1]. In the past ten years, NMR has made significant contributions to horticulture [2, 3], biotechnology, chemical engineering, petroleum science and food technology and now stands on the threshold of making an impact on environmental monitoring, building technology, and security

technology. Traditionally NMR is performed using laboratory or clinic based superconducting magnets, but now it is moving out into industry in the form of portable permanent magnet based systems. NMR development is being driven by advancements in electronics, computing and magnet technology and so continues to advance in capability and application.

2. Nuclear Magnetic Resonance

Certain nuclei possess an intrinsic angular momentum and magnetic moment and when placed in a magnetic field, B_0 , the nuclear moments will precess about the applied field direction at the Larmor frequency $\omega_0 = \gamma B_0$ (Fig. 1). The spins may be aligned or opposed to the field direction. At room temperature more spins are aligned than opposed with the field giving rise to a net magnetization vector, M , aligned with the applied field.

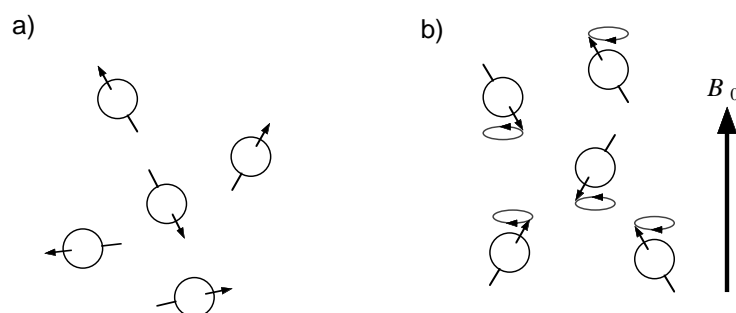


Fig. 1. Nuclear magnetic moments a) before and b) after the application of a magnetic field.

We interact with this magnetization by applying short radio frequency (RF) pulses at the Larmor frequency to a resonator surrounding the sample. The magnetic component of this pulse, B_1 , causes the magnetization to rotate away from the z (B_0) axis into the x - y plane (Fig. 2). Once the pulse has finished the magnetization will continue to precess about B_0 at ω_0 . The same resonator which produced the B_1 pulse can now be used to detect the EMF induced by the precessing magnetization. This induced signal, called the free-induction decay or FID, has a level ranging from μV to mV and takes the form of a decaying sine wave. The decay arises from relaxation processes which cause the perturbed magnetization to return to its equilibrium position. The characteristics of the relaxation provide insight in to the molecular environment within the sample and the dynamic processes taking place.

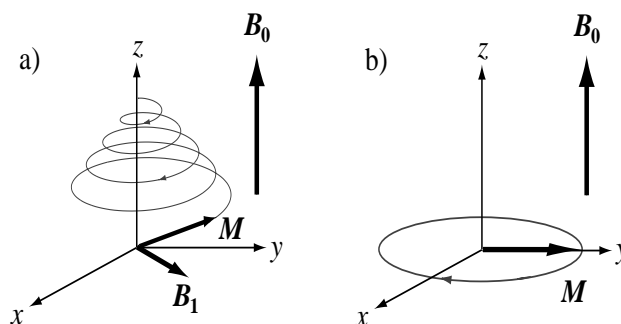


Fig. 2. a) During the B_1 pulse the magnetization spirals down about the z axis until it ends up in the x - y plane; b) Following the B_1 pulse the magnetization precesses freely in the x - y plane at frequency ω_0 .

Superconducting magnets have been the key to the success of laboratory based NMR systems, however they can cost millions of dollars to purchase, are very expensive to maintain and require special facilities to house them. But the information that NMR can obtain makes them invaluable. Nuclei are affected by other surrounding nuclei which gives rise to a distribution of resonant frequencies. This property is the heart of NMR spectroscopy and allows the determination of nuclear structure. By applying additional magnetic field gradients, spatial information can also be obtained. This is the basis for Magnetic Resonance Imaging (MRI).

3. Portable NMR Systems

NMR is now moving out of the laboratory and into the field in the form of low cost permanent magnet based systems [10]. They are limited in performance when compared to laboratory systems but nevertheless are still very useful tools for specific applications. Every NMR system whether laboratory based or portable basically consists of a probe, a set of supporting electronics and a user interface for controlling the system and observing/collecting the data. The block diagram for such a system is pictured in Fig. 3.

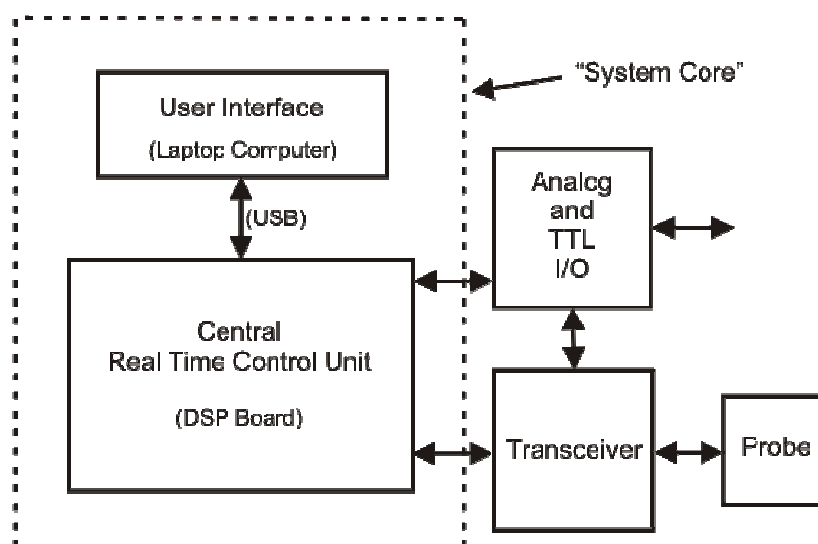


Fig. 3. NMR System Architecture.

3.1. Portable NMR Probes

Probes come in a range of sizes and designs and are the critical interface between the material that one wants to study and the user. The performance of the entire system is largely dependent upon the probe.

3.1.1. The NMR “MOUSE”

The **Mobile Universal Surface Explorer (MOUSE)** probes, developed by a group in Aachen, Germany [4] are based on the principles of “inside-out” NMR where the region of interest is external to the probe. They were initially developed for analyzing rubber and plastics, but have now been applied to areas such as the moisture determination of historic documents and buildings. The NMR-MOUSE is a surface probe that can only be used to obtain data from samples that are located within a few mm of the probe surface. Fig. 4 gives a schematic and constructed view of the hand held probe. Two

rectangular neodymium-iron-boron permanent magnets are placed on an iron yoke to generate the necessary B_0 field that is aligned with the surface of the probe. A solenoidal B_1 coil is used to interact with any sample that is placed near the probe surface.

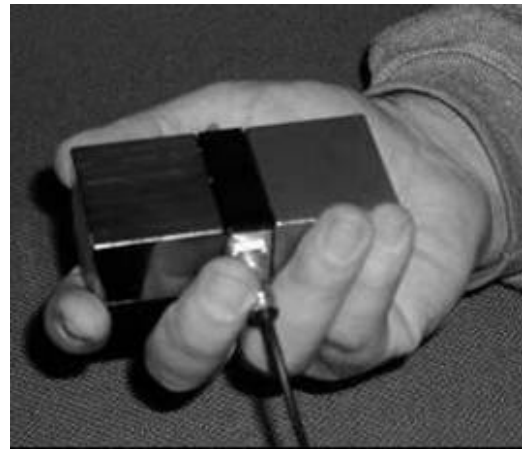
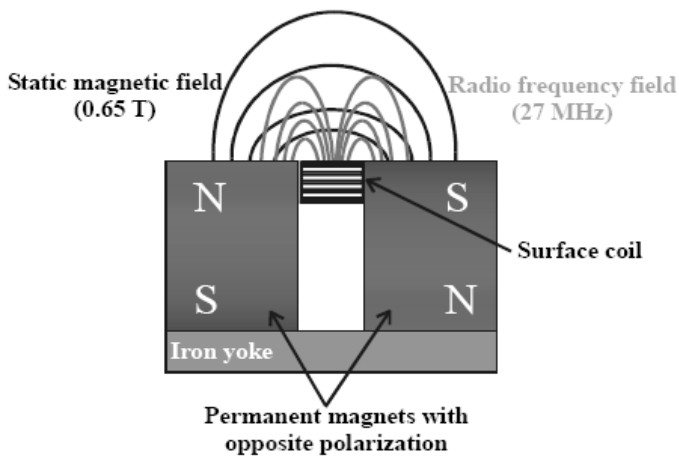


Fig. 4. Schematic of NMR-MOUSE, magnet and B_1 coil configuration (left), constructed NMR-MOUSE (right) [4].

An alternative to this design is the handheld single cylindrical bar magnet MOUSE shown in Fig. 5 [4, 9]. Here a “double D” B_1 coil is placed directly on the end surface of a cylindrical magnet and is designed to generate a field parallel with the surface of the probe. This is the opposite to the configuration of the earlier two magnet MOUSE design. Again only samples within a few mm from the surface can be measured.



Fig. 5. The handheld single bar magnet MOUSE. The field strength at the surface is approximately 0.46 T, equating to a proton resonance frequency of 19 MHz. The strength of the alternating field produced by the B_1 coil for 100 W of RF power is 1.5 mT and the typical B_1 pulse duration is 4 μ s [4, 9].

3.1.2. The NMR “MOLE”

What is required for applications such as the monitoring of drying concrete is a probe that can obtain data from deep inside the material. A probe called the NMR-MOLE was designed to produce a one

cubic centimeter region 10 mm into the sample (Fig. 6) [5, 8]. A series of individual magnets were used to approximate a ring magnet and a further magnet was placed in the centre. The position of central magnet and the angle and positions of the ring magnets were adjusted until the desired field profile was achieved. The probe has a diameter of 250 mm, field strength of 65 mT at the sweet-spot and weighs approximately 6 kg. The B_1 coil was constructed using a printed circuit board (Fig. 7) and produces a $250 \mu\text{T}$ field in the sample region for 100 W of RF power. The typical B_1 pulse duration is $30 \mu\text{s}$.



Fig. 6. (a) The configuration of magnets used to produce a homogenous region [5, 8]. (b) The complete prototype NMR-MOLE probe. The magnets are placed into a plastic housing and the B_1 coil is placed onto the surface as shown.

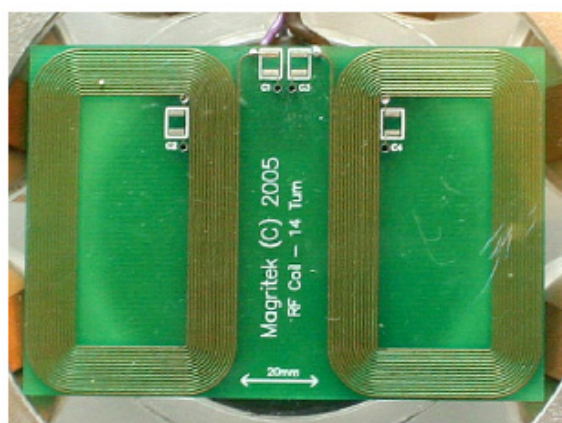


Fig. 7. NMR-MOLE B_1 coil, a variation of the “double D” coil design [8].

3.2. System Electronics

NMR like many other techniques requires the stimulation of a sample and then the monitoring of any emissions. It is analogous to the impulse response technique often used to characterize electronic systems. A radio frequency pulse is applied to a coil close to the sample and the subsequent Free Induction Decay (FID) signal is received by the same coil, and after amplification, is acquired. For permanent magnet systems the stimulus/emission is often in the tens of MHz region so radio frequency transceiver techniques are required.

3.2.1. System Core

What is common to all NMR applications is the generation of precisely timed signals, the capturing of FIDs and the processing/display of data. Most of this has been encapsulated into a single unit known as the system core [6], see Fig. 3. This is based around a general purpose Digital Signal Processor (DSP) and a Universal Serial Bus (USB) interface that is used to communicate with a host laptop computer. A graphical user interface is provided by an application running on Windows XP. This interface is fully configurable and scriptable through the use of a macro language and a set of standard commands. Pulse programs can be generated using the built-in language provided by the user interface or by using a C compiler/assembler to generate code for the DSP itself. The DSP runs at 100 MHz and therefore provides a pulse program timing resolution of 10 ns. A block diagram of the system core is shown in Fig. 8.

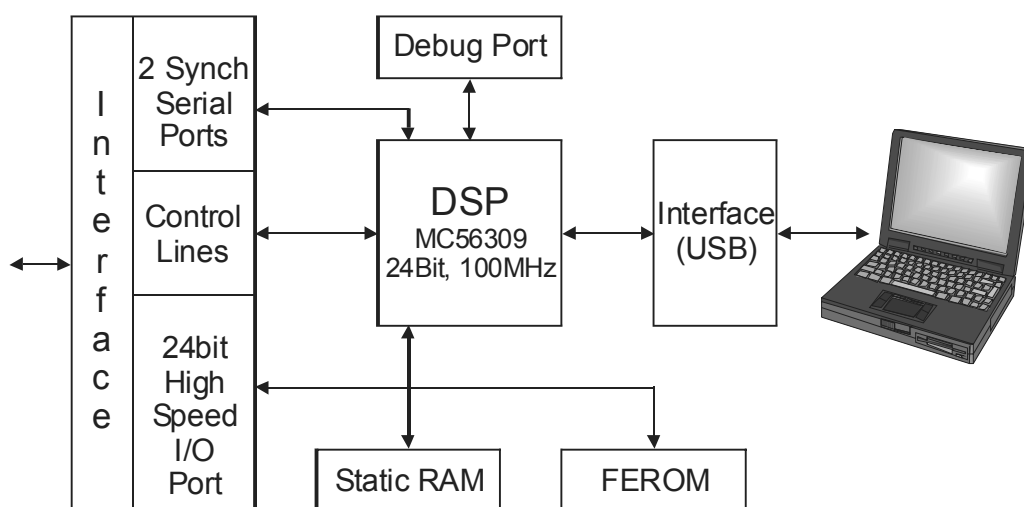


Fig. 8. The system core consisting of a DSP board that uses USB to interface to a host Laptop computer.

3.2.2. Digital Transceiver

The RF section of a NMR system is very similar to a communications transceiver. A major advancement in recent years has been the introduction of digital transceiver technology. One example is the AD6620 from Analog Devices [7]; a simplified block diagram is shown in Fig. 9. Here the received signal is sampled either after an IF stage or directly after the preamplifier. The sampled signal is then mixed digitally with synthesized sine and cosine functions to generate the lower frequency quadrature outputs. Digital filtering is then applied to reduce the bandwidth and finally down-sampling is used to reduce the output data rate. A digital transceiver was developed based on the AD6620. A block diagram is shown in Fig. 10.

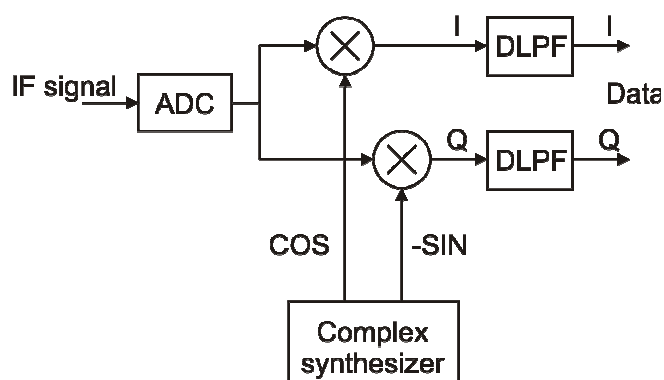


Fig. 9. The AD6620.

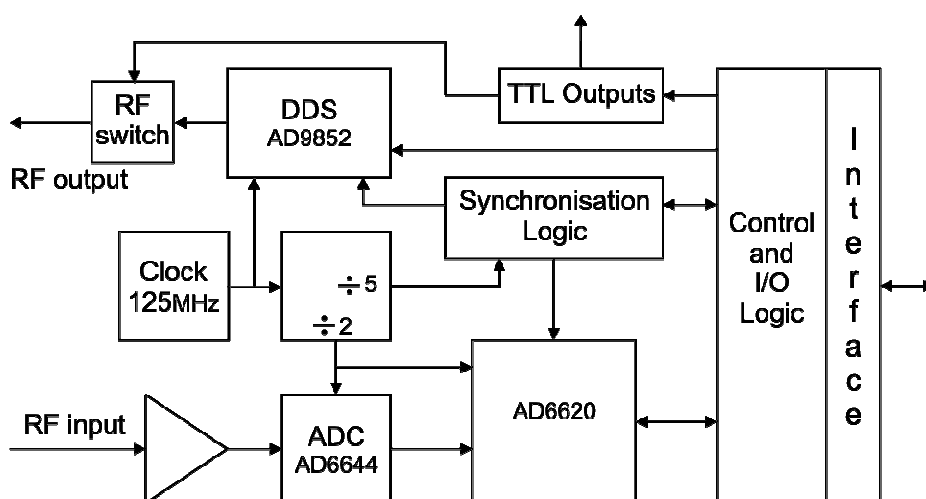


Fig. 10. Digital transceiver.

The AD6644 14-bit ADC samples at a rate of 62.5 MHz and sends its digital values directly to the AD6620. The output of the AD6620 receiver is fed to the system core DSP for further processing and storage. An AD9852 Direct Digital Synthesizer (DDS) is also included to generate any required B_1 signals. Both the synthesizer and the AD6620 NCO have phase and frequency hopping capabilities and are phase locked to each other and the DSP. The DSP/USB unit and RF transceiver are each built on standard Euro-card sized multi-layer PCBs (Fig. 11) and connect to each other via a back plane.

3.2.3. RF Electronics

To complete the NMR spectrometer system a RF front end was developed (Fig. 12) consisting of a high power (100 W) broadband linear RF amplifier, a very sensitive low noise preamp and a fast transmit/receive switch.

A directional coupler is used for tuning and matching the probe coil to 50Ω , diodes D_1 block any residual noise from the power amp during receive and diodes D_2 together with a lumped element quarter wavelength transmission line act as a transmit/receive switch to protect the preamp during

transmission. During transmission all the diodes conduct, therefore diodes D_2 present themselves as a short circuit to the quarter wavelength transmission line which in turn appears as high impedance to the RF power source resulting in minimal power going into the very delicate preamp. Typically the transmit pulse duration ranges between 2 and 40 μ s. A broadband variable gain amplifier was designed to amplify the signal from the preamplifier up to a level that is suitable for the digital receiver input.

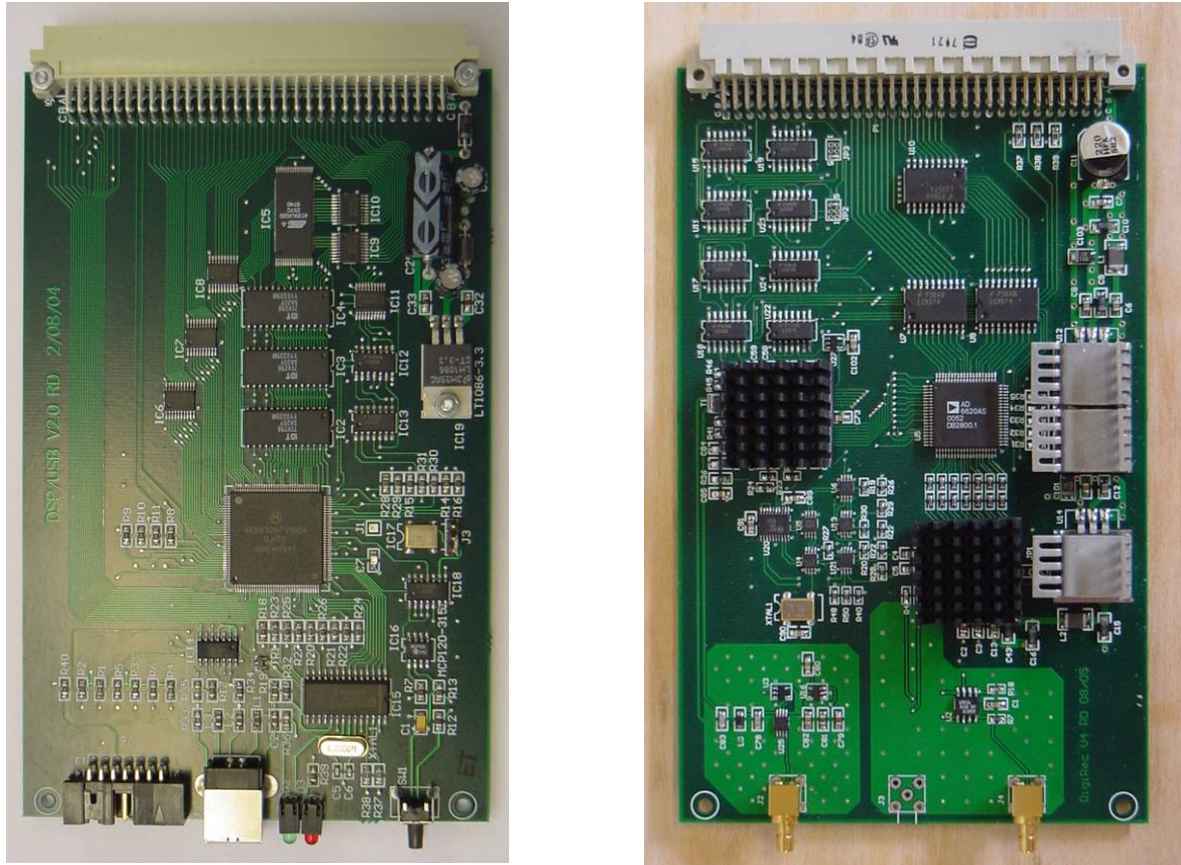


Fig. 11. DSP (left) and digital transceiver (right) PCBs.

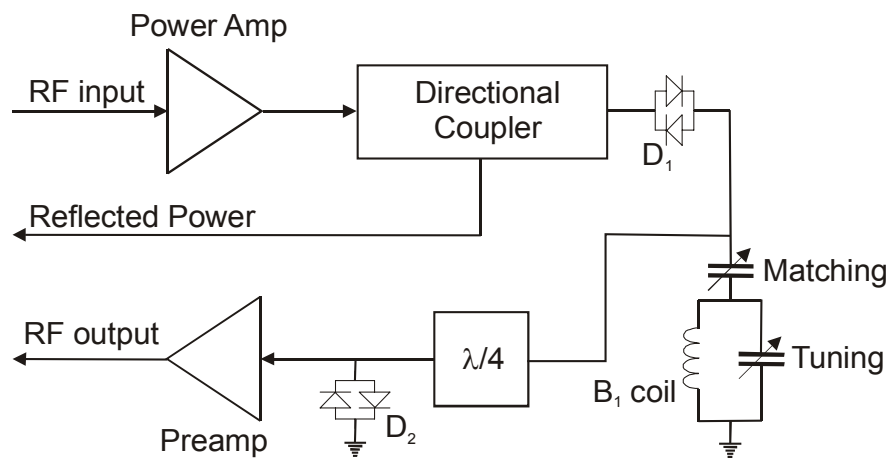


Fig. 12. RF front end.

3.2.4. Power Supply and Enclosure

A switchmode power supply module was developed to enable the entire NMR system to be powered from a battery. The complete NMR spectrometer consisting of the DSP/USB unit, the digital transceiver, the variable gain amplifier, the RF front end and the power module was housed in a special compact enclosure to make the system portable and is shown in Fig. 13.



Fig. 13. Complete battery powered portable NMR system. The spectrometer part measures 310x230x55 mm and weighs 3.6 kg. The battery pack shown can provide 3 to 4 hours of operation, depending on the RF power requirements.

3.2.4. System Testing

The spectrometer was first used with a single bar magnet MOUSE and a rubber sample to verify that the system was operating correctly. The top part of Fig. 14 shows an echo signal obtained using 10 experiment averages. Some time after the first RF pulse, another RF pulse is applied to refocus the dephasing magnetization to form an echo. The amplitude of the echo is directly proportional to the number of protons in the sample. This refocusing idea can be repeated many times so that a series of echoes can be obtained and this is shown in the bottom part of Fig. 14. This is called a Carr-Purcell-Meiboom-Gill, (CPMG) sequence and is used to obtain information about relaxation processes that are occurring within the sample material. The decay can tell us about the molecular environment of the protons and therefore give us information about some of the material properties.

4. Experiments

The first example for the potential use of portable NMR is shown below (Fig. 15) for a small (20 ml) gelating pectin sample, measured on the NMR-MOLE probe using a CPMG pulse sequence. As the viscosity of the gel increases with time, movement of water molecules becomes increasingly restricted leading to a reduced spin-spin (T_2) relaxation time.

Another example of the potential use of portable NMR is in the dairy industry. Both cheese and butter begin with milk, a food containing mostly water but with appreciable amounts of fat, protein, lactose and minerals. Applying different processes to the milk produces many different varieties and kinds of butter and cheese. Typically, butter contains about eighty percent milk fat and fifteen percent water, while cheese contains five to ten times concentrated milk but with most water removed, leaving about forty percent bound water.

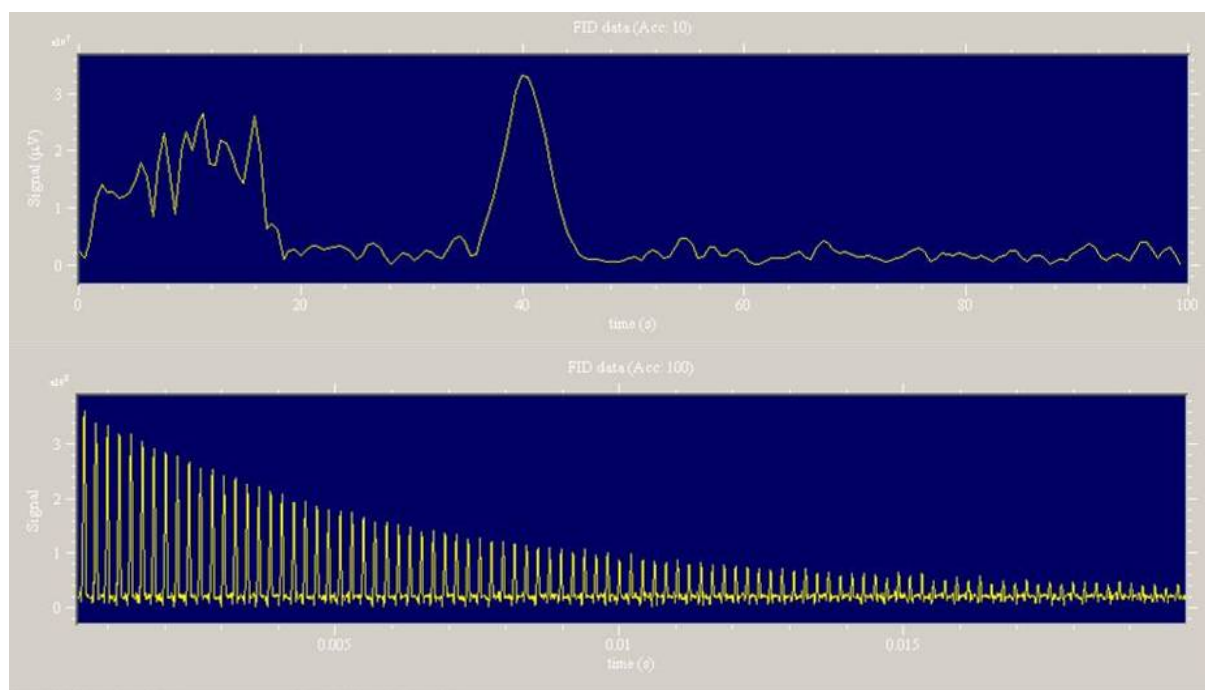


Fig 14. The top trace is the signal obtained using a Spin Echo experiment with a bar magnet NMR-MOUSE and a rubber sample. To improve the signal to noise performance, ten experiment scans were performed and the data averaged. The middle part of the screen shows the echo received from the sample. The bottom trace shows the data obtained using a CPMG experiment, where multiple refocusing pulses are applied to produce a series of echoes.

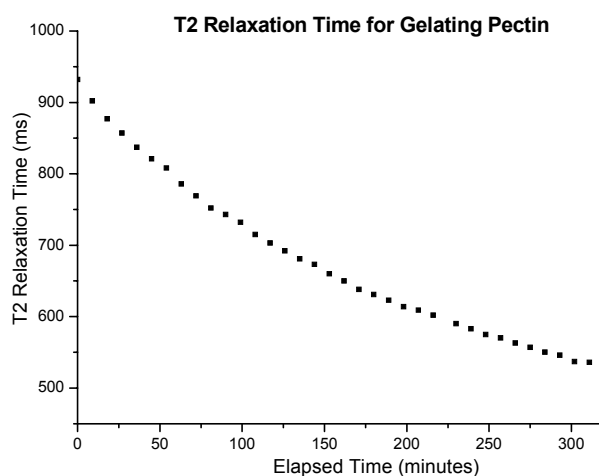


Fig. 15. Gelating pectin solution. Gel viscosity increases with time, resulting in shortened relaxation time as motion of the water molecules becomes more restricted.

Figs. 16 and 17 contain measurements of T2 relaxation times using a CPMG sequence, averaged over 128 scans. In each case, the left pane shows the time-decaying echo amplitude envelope, and the right pane shows the relaxation spectrum which is an estimation of the distribution of frequencies contained in the envelope, determined using a Laplace inversion technique. Fig. 16 shows data from *Rolling Meadow* butter. The relaxation spectrum has three peaks; the larger centered at 29 ms with amplitude 7.4 units, the second smallest at 130 ms with amplitude 2.0 units and the smallest at 600 ms with amplitude 1.0 units. The relative amplitudes of the peaks are approximately seven-to-two-to-one. From the known properties of butter it is most likely that the seven units relates to seventy percent of the milk fat which is in crystal form, one of the smaller peaks makes up the other ten percent milk fat in globule form, and the remaining peak is the fifteen percent water.

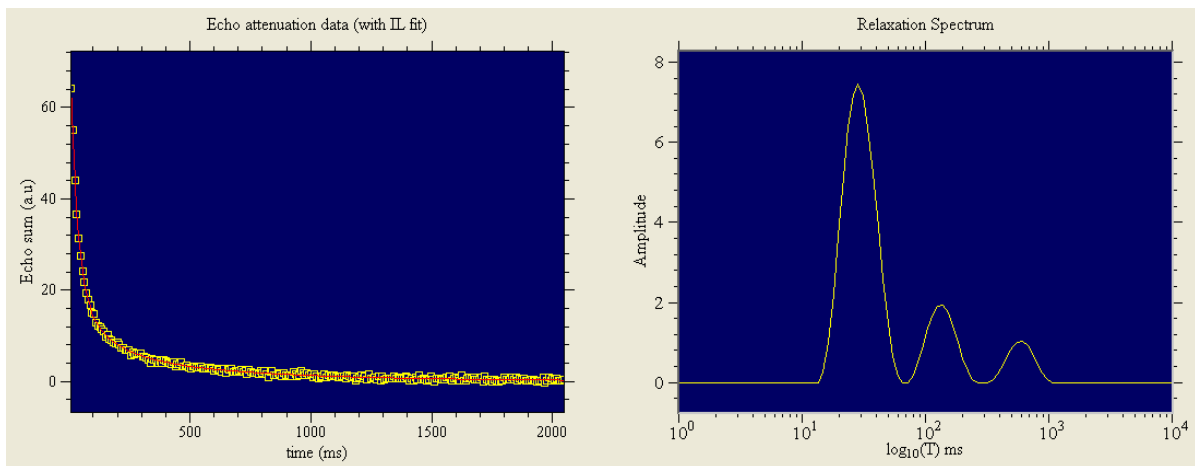


Fig. 16. CPMG Echo attenuation data and corresponding relaxation spectrum for butter.

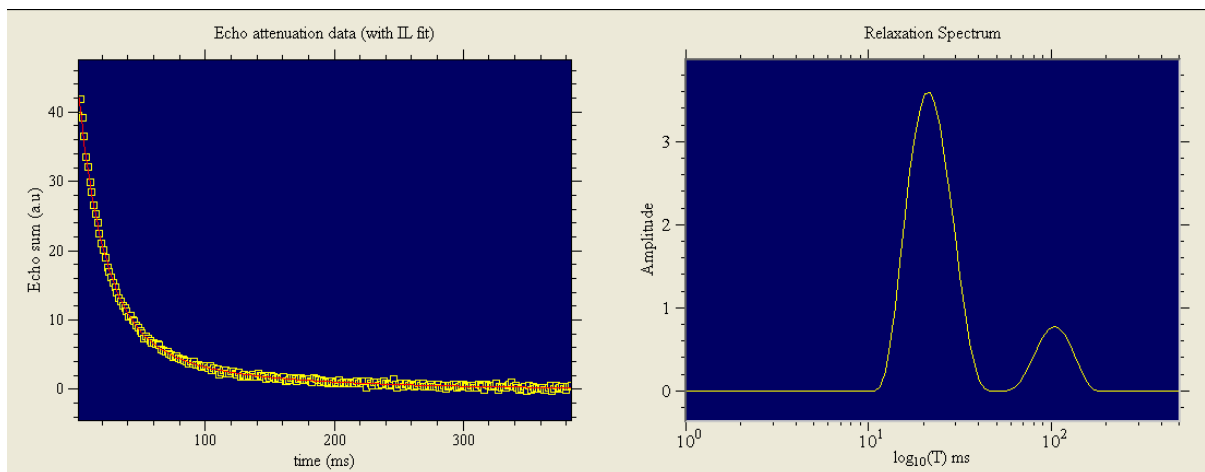


Fig. 17. CPMG Echo attenuation data and corresponding relaxation spectrum for cheese.

Fig. 17 shows data from *Dairymaid Colby Cheese*. The relaxation spectrum has two peaks; the larger centered at 21 ms with amplitude 3.3 units and the smaller at 100ms with amplitude 0.8 units. The manufacturer lists the moisture (bound water) as typically thirty seven percent by weight with fat making up the bulk of the remaining seventy three percent. The larger peak most likely corresponds to the fat and the smaller peak to the water.

5. Conclusions

A complete portable NMR system has been designed, constructed and verified and can be used with either a NMR-MOUSE or NMR-MOLE probe. Preliminary experiments verify the potential for this system to be used in the non-destructive testing of many materials. In principle, this system could be utilized in the measurement of fruit ripeness and fat content as well as other applications such as determining the molecular mobility in resins, rubbers or concrete.

Acknowledgements

The authors would to thank the technical staff from the science workshops of Massey University, Victoria University of Wellington and Aachen University of Technology. This project was made possible by funding provided by the New Zealand Foundation for Research Science and Technology.

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