Project Summary

Overview

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Project Description

DNP NMR signal enhancement is seen at high field, and high frequencies. The necessary high frequency GHz EPR signal imposes a unique set of electromagnetic design requirements [14]. For the sample to get the necessary amount and kind of magnetic fields for DNP, several design requirements must be met. The EPR frequency part of the system is comprised of some waveguide carrying the 527 GHz microwaves, a transition structure, and the sample cavity. The waveguide should exhibit a minimum of loss. The coupler must deliver magnetic fields to the sample transverse to the main field, as homogeneously distributed as possible. The sample cavity needs to be low loss resonator at both the EPR and NMR frequencies (a double resonator), that is, have a high loaded Q. A quantity known as conversion factor, the magnetic field strength per power applied, should be maximized. Further it must be strong enough to withstand the high pressure (70 bar) associated with using supercritical CO2 as a solvent [26]. This part of the proposal describes the design choices and work plan of the coupler and sample cavity, since the waveguide exists already. Given the unique electromagnetic and structural requirements of our system , it is likely we will have to develop a new approach for the coupling and resonator. There are several major design choices: resonant structure, material, and orientation.

First, we must determine the EPR resonator geometry. The resonator has to achieve resonance at the EPR frequency while preserving resonance at the NMR frequency using YBCO [5] NMR coils on sapphire substrate. Additionally, the magnetic fields must be transverse to the main field as well as being as homogeneous as possible. The desireable modes in the resonator depends on orientation of sample - in a traditional cavity resonator, transverse to main field, we need TE_{011} in order to have the magnetic field transverse to the B_0 field [22, 30], while if the sample is coaxial, we need TM_{110} [14]. In a quasi-optical resonator, transverse to the main field, a low-order TEM mode may be used [6].

A traditional approach in electromagnetics would be to use a dielectric resonant cavity. Used as antenna, it can support radiating modes, though that's not desirable here [20]. [15] uses a cylindrical dielectric resonator at 150 GHz, with the long axis perdendicular to the main field, holding a 0.7 inside diameter sample bulb. That instance supports the TE_{011} mode, where there is no B_z component. At 90 GHz, [1, 2] used a u-shaped NMR coil looped inside a dielectric tube TE_{011} resonator formed by a quartz sample capillary of 0.8mm ID. It ran perpendicular through two metal mirrors, serving as a planar waveguide supporting a TE_1 mode delivered through a rectangular waveguide. A TE_{011} 12.7 mm by 2.7 mm metal cavity resonator made using a helical tape NMR coil formed a TE_{011} resonator, with adjustable metal plungers on each end to adjust the resonance. The center of the resonator held a 0.5mm ID quartz capillary. It is excited by 139.5 GHz waves through a slot aperture connected to a waveguide.

Other traditional electromagnetic structures function as resonators. [21] built a planar THz time domain spectroscopy resonator operating around 275 GHz, which functioned as a flow cell. It contained an 8 μl groove, carrying a TE_1 mode. This is an interesting design considering our requirement of s-CO2 solvent, ill-suited for a traditional quartz capillary. [32] constructed a laser-tunable bandpass filter of high temperature superconducting YBCO crosses on MgO substrate. Printed ring shapes formed optical resonators at λ of 1 μm in a channel drop-add filter [19]. Stripline NMR [27], where the NMR B_1 is supplied by the current though a planar strip, is a type of resonator. It operated at 600MHz but could be used in a dual-resonance structure [6]. [6] and others have used quasi-optical resonators, which are designed using ray optics. In particular, Fabry-Perot quasi-optical resonators have seen use in EPR [6] and masers [9]. In the former, the resonator is formed by a semi-confocal spherical mirror reflecting the EPR field (260 GHz) onto a planar mirror formed by a planar stripline NMR probe (400 MHz) holding a 80 nl sample. Generally they are formed by two mirrors facing eachother, excited by a small aperture, though multiple mirrors can arranged in a ring [23].

Second, the optimal cavity material - it should be low loss, have an appropriate dielectric constant, and high strength. The type or resonator will govern and thus it's structure will determine the exact necessities. If treated as a resonant dielectric cavity, to achieve resonance, it requires a high dielectric constant around the sample for a dielectric resonator [4]. That's not necessarily required for a quasi-optical resonator. Additionally, the material should be low loss at both NMR and EPR frequencies, and be nonmagnetic and free of radicals. [18] compiles at list of THz properties of materials; teflon [13], HDPE [16], sapphire and silica [28] have good properies. Since the resonator must contain the sample solvated in s-CO2, it must be strong, with a high modulus of elasticity and high ultimate strength [7, 8, 11].

Finally, we need to determine optimal orientation of waveguide (transverse or parallel to main field) and sample (transverse or parallel to the main field). Related to this is the choice and design of coupler. In each case, electromagnetic modes in waveguide, and the allowable modes in the resonator, determine nature of coupling. In quasi-optical wave guides these are Gauss-Hermite or Gauss-Laguerre plane wave TEM single-mode [13, 16] or TE, TM modes in traditional metal waveguides of small dimension [10]. The relative orientation of the waveguide and cavity, as well as the cavity resonant modes, determines the type of coupler. In quasi optical resonators, such as Fabry-Perot, an iris in the confocal reflector, fed coaxial to the main field, works [6] or alternatively, a quarter wavemength transformer following a horn [25]. A parabolic reflector can transform the TEM modes in a quasi-optical waveguide to a transverse mode with a different orientation [31]. Alternatively, traditional means of coupling include apertures of differing dimensions to excite different modes in a cavity resonator [4].

To analyze the problem, the initial step is purely theoretical, making use of either a tradtional cavity resonator approach [4], a solution directly of Maxwell's equations or, because we are operating at sub-millimeter wavelengths quasi-optical [12]. This approximates wave equation with parabolic wave equation and uses ray techniques of optics, assuming a Gaussian beam. Elements in the structure are represented by matrix operators on the ray. Different components can be treated as simple ray matrix operators, such as diplexers [3], waveguides [10], and filters [12, 29]. Since we make use of a quasi-optical waveguide, this approach makes more sense. Quasi-optical analysis is a smart approach if we use a quasi-optical resonator, in order to ensure stability [17, 24]. Of course, if examining a tradtional cavity resonator for the EPR, or ensuring we will have resonance at the NMR frequency in the NMR coils, we will use a traditional, full-wave analysis of a resonant cavity. Such an analytical approach first will help choose the best type and orientation of resonator. We will evaluate all four possible orientations of sample and waveguide analytically for EM suitability, and choose the one which should produce the optimal fields, in terms of resonance and homogeneity, while maintaining structural integrity under high pressure. After selection, a finite-element multiphysics simulation will help refine the chosen design, particularly to account for the strain imposed by the high-pressure s-CO2 and the dielectric loss and heating.

Once we have a resonator and coupler design, the next steps are to fabricate and test the coupler and resonator. Fabrication will depend on the precise geometry and materials, but could likely be accomplished at one of UF's machining shops. The resonantor would need to be tested first on a vector network analyzer to establish that it resonates at the frequencies of interest, and evaluated with field probes to assure magnetic fields at the EPR frequency are transverse to the B_0 field. Additionally, the resonator should be tested as a pressure vessel for s-CO2, before it is finally tested as a system inside the magnet. At that stage we will verify basic functionality, checking that there is an NMR signal response to application of EPR pulses, and that there is a response to changes in input power. Further testing will establish the exact sensitivity benchmarks associated with high field NMR.

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Results From Prior NSF Support

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Biographical Sketch: Your Name

(a) Professional Preparation

(b) Appointments

(c) Products

(d) Synergistic Activities

Data Management Plan

Collaborators and Other Affiliations Information

Collaborators and Co-Editors

Graduate Advisors and Postdoctoral Sponsors

Thesis Advisor and Postgraduate Scholar Sponsor