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Albannay, Mohammed M.; Vinther, Joachim M.; Zhurbenko, Vitaliy; Ardenkjær-Larsen, Jan H.

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Low microwave attenuation and low thermal loss waveguides for dDNP probes

Mohammed M. Albannay1, Joachim M. O. Vinther2, Vitaliy Zhurbenko1, Jan H. Ardenkjær-Larsen1,2
1Center for Hyperpolarization in Magnetic Resonance (HYPERMAG), Department of Electrical Engineering, Technical University of Denmark, Kgs Lyngby, Denmark. 2GE Healthcare, Brønby, Denmark.

Microwave sample irradiation is essential to perform DNP. Waveguides provide an effective way of coupling the output of a microwave source to the electron spins. Individually, the waveguide introduces a significant thermal heat load into the sample space of our dDNP probe. The use of a circular stainless steel waveguide with an internally electroplated layer of copper offers an effective, economical solution to address this problem.

**dDNP probe**

Microwave irradiation is a requisite to transfer electron spin polarization to nuclear spins. Significant increase in NMR sensitivity by way of dissolution DNP (dDNP) [1] has encouraged the development of multiple commercial and home-built polarizers and dDNP probes [2-3].

**Engineering challenge**

The length of waveguide needed to couple a microwave source to the electron spins is dictated by the dimensions of the polarizer, thereby influencing the total waveguide attenuation. The desire for higher magnetic fields (B0) has raised the required microwave frequency to perform DNP, further limiting the available power due to inefficient solid-state microwave sources.

Corrugated waveguides improve microwave irradiation by reducing transmission losses, but are costly to procure [4]. Similarly, mode converters offer use of propagation modes with reduced attenuation constants, but are challenging to fabricate at higher frequencies and have some insertion loss. We present a solution to achieve efficient microwave irradiation whilst minimizing thermal loss.

**DNP-NMR experiments**

The effect of polarization using a stainless steel and copper plated waveguide was investigated using a 100 µL 4.5 M [13C]urea (5:4:1 glycerol-d5, D2O, H2O & 40 mM TEMPO) sample in a 6.7 T polarizer. 2H polarization was observed using low flip angle pulses.

The sample was irradiated with 188.06 GHz microwaves having a frequency modulation bandwidth of 50 MHz with a frequency of 1 kHz. No chamfers or reflectors are employed in the overmoded cavity. Methods to improve irradiation efficiency are currently being explored.

**Microwaves in DNP**

**Thermal conduction vs. attenuation**

The probe is permanently equipped with a waveguide, coupling the top flange to the cryogenically cooled sample space. The conducted thermal heat decreases with the waveguide’s cross-sectional area therefore a 4.16 mm circular stainless steel waveguide was selected since it offers the lowest attenuation for a given perimeter (when compared to a rectangular waveguide). Ohmic losses are reduced by internally electroplating the waveguide with a layer of copper.

**Waveguide electroplating**

Solutions are pumped through the waveguide using a peristaltic pump. Once coated they are mechanically polished to reduce surface roughness, resulting in a shiny pink finish. The waveguide is rinsed and then dried with an inert gas.

**Waveguide measurements**

Waveguide attenuation was measured using a 94 GHz source and a doubler or tripler. The reliability of the measurements were improved using an anti-cocking UG387 adapter and an alignment flange.

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