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

Publication date:
2018

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):
Low microwave attenuation and low thermal loss waveguides for dDNP probes

Mohammed M. Albannay¹, Joachim M. O. Vinther¹, 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, Brøndby 2605, 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. Indireptly, 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**

**Microwaves in DNP**

Microwave radiation 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.

C 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 [13]C]urea (5:4:1 glycerol-d₃, D₂O, H₂O & 40 mM TEMPO) sample in a 6.7 T polarizer. °H 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.

**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 e4.16 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.

**Acknowledgement**

This work was financially supported by the European Union’s Horizon 2020 research and innovation programme under the Marie Skłodowska-Curie grant no. 642773 and the Danish National Research Foundation grant no. DNRF124

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