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Publication date:
2018

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

Citation (APA):
Albannay, M. M., Vinther, J. M., Zhurbenko, V., & Ardenkjær-Larsen, J. H. (2018). Low microwave attenuation and low thermal loss waveguides for dDNP probes. Poster session presented at International Conference on Nuclear Hyperpolarization 2018, Southampton , United Kingdom.

Low microwave attenuation and low thermal loss waveguides for dDNP probes

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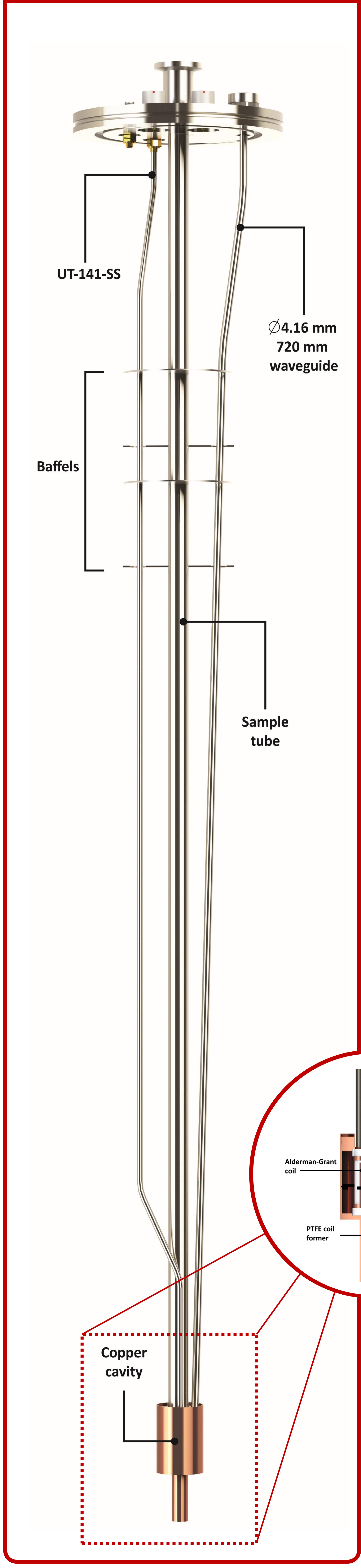
Microwave sample irradiation is essential to perform DNP. Waveguides provide an effective way of coupling the output of a microwave source to the electron spins. Indivertibly, the waveguide introduces a significant thermal heat load into the sample space of our dDNP probe. The use of a circular stainless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

dDNP probe

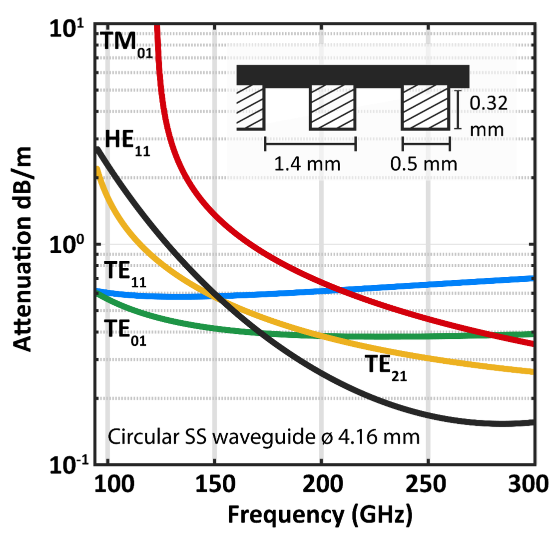


Microwaves in DNP

Microwave 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.



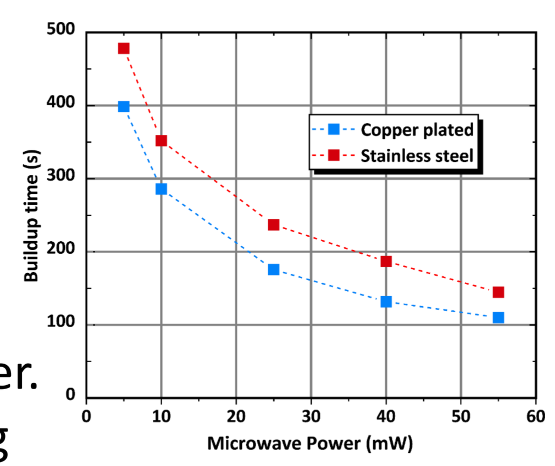
The desire for higher magnetic fields (B_0) has raised the required microwave frequency to perform DNP, further limiting the available power due to inefficient solid-state microwave sources.

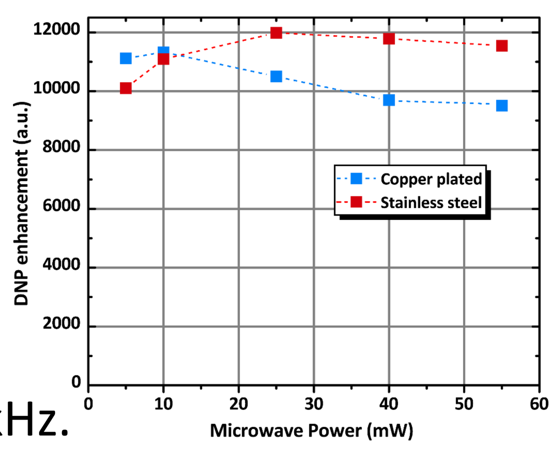
Corrugated waveguides improve microwave irradiation by reducing transmission losses, but are costly to procure [4]. Similarly, mode converters offer use of propagation modes with reduced attenuation constants, but are challenging to fabricate at higher frequencies and have some insertion loss.

We present a solution to achieve efficient microwave irradiation whilst minimizing thermal loss.

DNP-NMR experiments

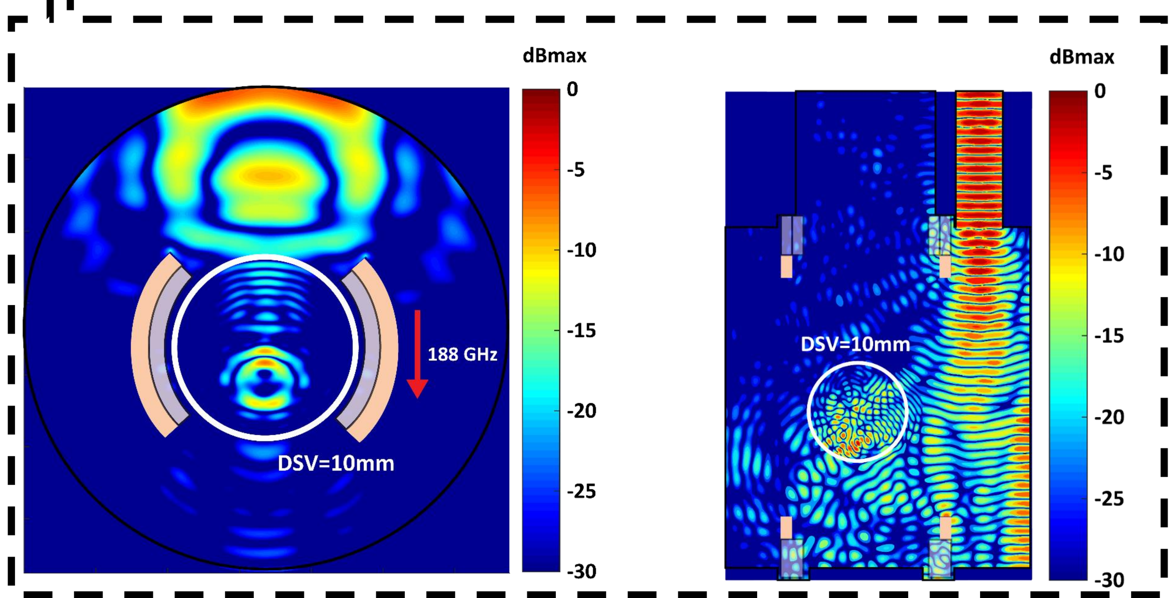
The effect of polarization using a stainless steel and copper plated waveguide was investigated using a 100 µL 4.5 M [^{13}C]urea (5:4:1 glycerol-d_8, D_2O, H_2O & 40 mM TEMPOL) sample in a 6.7 T polarizer. ^{1}H polarization was observed using low flip angle pulses.





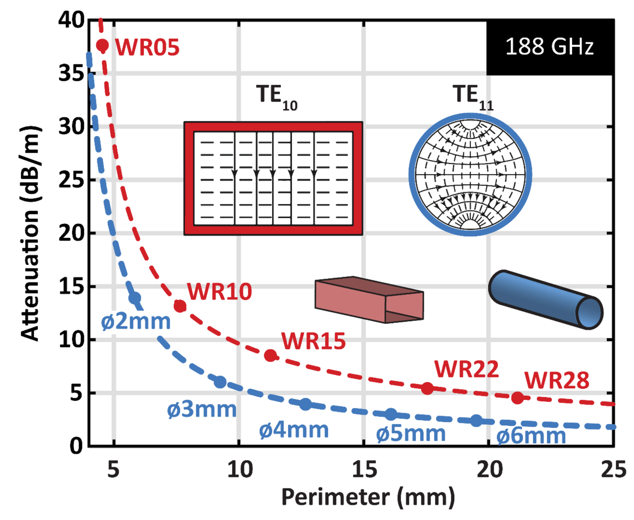
The sample was irradiated with 188.06 GHz microwaves having a frequency modulation bandwidth of 50 MHz with a frequency of 1 kHz.

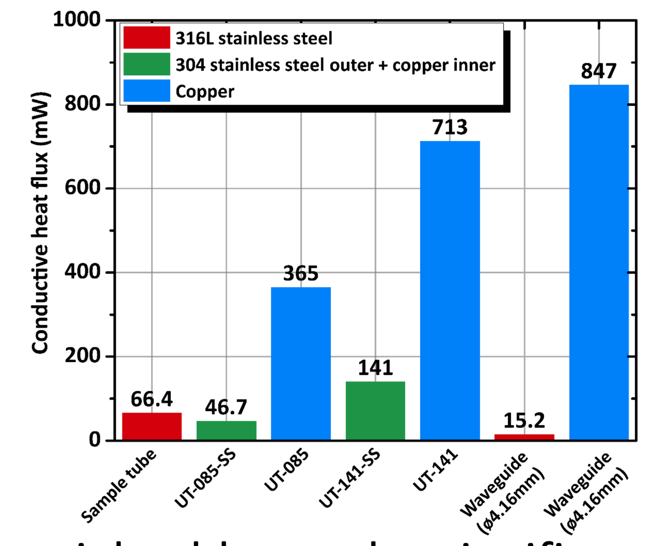
No chamfers or reflectors are employed in the overmoded cavity. Methods to improve irradiation efficiency are currently being explored.



Thermal conduction vs. attenuation

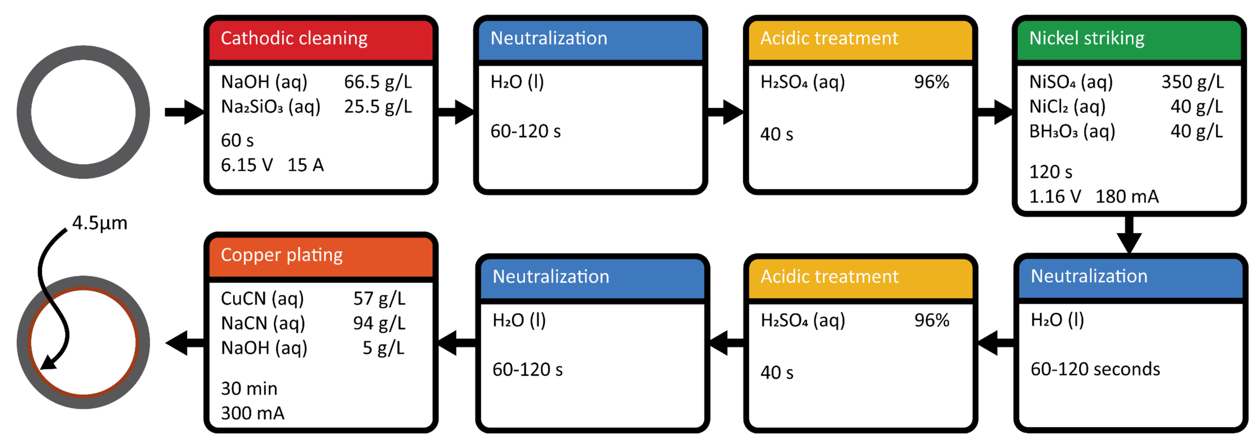
The probe is permanently equipped with a waveguide, coupling the top flange to the cryogenically cooled sample space. The conducted thermal heat decreases with the waveguide's cross-sectional area therefore a ⌀4.16 mm circular stainless steel waveguide was selected since it offers the lowest attenuation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





Thermal heat coupled by a coaxial cable can be significant (particular via the inner conductor). A compromise between electrical loss and thermal loading can be achieved using a cable with a stainless steel outer conductor.

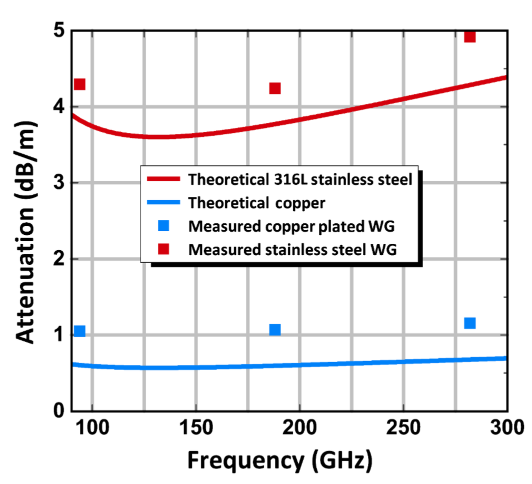
Waveguide electroplating

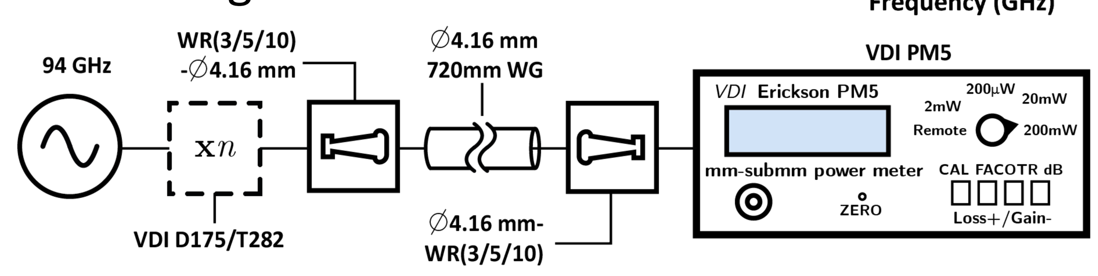


Solutions are pumped through the waveguide using a peristaltic pump. Once coated they are mechanically polished to reduce surface roughness, resulting in a shiny pink finish. The waveguide is rinsed and then dried with an inert gas.

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 Sklodowska-Curie grant no. 642773 and the Danish National Research Foundation grant no. DNRF124

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