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Discussions

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Reply on RC2

Aaron Himmler et al.

Author comment on "Electroplated waveguides to enhance DNP and EPR spectra of silicon and diamond particles" by Aaron Himmler et al., Magn. Reson. Discuss., https://doi.org/10.5194/mr-2022-15-AC2, 2022

Thank you for the helpful comments and the carefull reading of the manuscript. Here is the point-by-point response to the comments:

Line34: it is stated that the “favorable corrugated waveguides are challenging to combine with a low temperature DNP cryostat”.
But, there are some successful examples in the referred literature describing EPR and DNP spectrometers with low temperature probeheads equipped with custom-tailored corrugated waveguides manufactured by Thomas Keating Ltd (UK) with very low transmission losses (0.1 dB/m) at frequencies above 180 GHz. Moreover, EPR/ENDOR probeheads (also equipped with a corrugated waveguide and compatible with Oxford Spectrostat) of the Bruker E-780 spectrometer operating at 263 GHz are commercially available on the market. Frame of the probehead could be suitable with your setup after minor change in the taper. Besides, some corrugated waveguides fabricated by GYCOM Ltd (Russia) which are made of German silver can also be a cheap option in case of a very limited budget. Please justify your choice more clearly.

A: Same answer as to reviewer 1:
We have no first-hand experience with corrugated wave guides and can only speculate. But we noticed that there is, to the best of our knowledge, no dissolution DNP system that uses corrugated wave guides all the way to the cryostat but just in connection with a quasi-optic transmission.
We believe the main reason corrugated waveguides are not commonly used in dissolution DNP, is the larger heat transfer due to the higher mass of such waveguides. However, we have not found any data for this. A second reason might be degradation due to moisture freezing in that changes the geometry but this could probably be solved by proper engineering.
Since we have no first-hand experience and data on these two points, we have modified the paragraph to a more cautious statement: "Gyrotrons are established systems to generate high microwave intensities (Blank et al., 2020) commonly used in MAS DNP systems but due to their much higher cost not a viable solution for dissolution DNP. Transmission of the microwaves to the sample space can be achieved by waveguides or corrugated waveguides with low losses (Nanni et al., 2012; De Rijk et al., 2011) or using quasi-optic transmission of the microwaves (Siaw et al., 2016). The desire towards implementing corrugated waveguides in low temperature DNP setups (Rijk, 2013; Leggett et al., 2010) shows that that minimizing transmission losses is an important strategy to maximize microwave power at the sample."

For us silver-plating was clearly the most simple solution to improve the power at the sample that did not require re-engineering of the probe. For a new design, we would probably think about a quasi-optic transmission of the microwaves to the sample space as an interesting alternative.

Line 103: “inner surface of the waveguide was first abrasively polished… then degreased with a water-based degreaser… ”.
Typically, stainless steel has a poor adhesion with electroplated metal layers such gold, silver, or copper, especially if the surface was not etched preliminary. It is not clear from the text how adhesion issue is fixed. Does the water-based degreaser work also as an etching agent, or do you accomplish an etching step before silver deposition additionally? If not, please comment on the adhesion issue.

A: No etching of the surface was performed prior to electroplating. To avoid adhesion problems, the gold layer was used in a first step. Experimentally, we have seen no problems with the adhesion of the layers since producing the first plated waveguides about a year ago. To make this clear, we added a short sentence around line 120: "No surface etching was required."

Line 156: “…in attenuation, namely 1.2 dB/m to 1.1 dB/m at 197 GHz…” The values look appropriate. However, oversized waveguides can transmit higher order modes beside the fundamental mode causing some standing waves along the oversized waveguide with tapers on both sides: the larger waveguide aperture the more propagating modes causing more standing waves. Presence of these standing waves can deteriorate performance of the waveguide at certain frequencies. It can be estimated by measuring transmission losses in the frequency range of interest, namely in the range from 196.7 GHz to 197.7 GHz in your case. Please add a transmission loss versus frequency plot to see how good is performance of the waveguide in the full frequency range.

A: We have measured the microwave power across the frequency range.
The power shows some variations over the whole 192-202 GHz range. However, the curve is quite smooth in the most used frequency range 196-198 GHz. After inserting the long overmoded waveguide we see some small fluctuations in power (ca. +-3%) across the frequencies, while the overall power curve is not distorted.

We hope this response answers all the points raised.