

Magn. Reson. Discuss., referee comment RC1
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Comment on mr-2022-15

Kong Ooi Tan (Referee)

Referee comment on "Electroplated waveguides to enhance DNP and EPR spectra of silicon and diamond particles" by Aaron Himmler et al., Magn. Reson. Discuss.,
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The work presented by Himmler et. al. is definitely interesting and relevant to be published in the MR journal. The authors have identified a usually overlooked situation in the dissolution DNP setup, i.e., the normal microwave waveguide is not very efficient. The solution presented is simple and effective, and most importantly, can be easily performed on a lab bench. Nevertheless, there are some technical details that can be further elaborated on and explained for better readability. Overall, I would recommend the manuscript for publication after addressing my questions below.

In the abstract

- change 'Lamour' to 'Larmor'

-Depending on the EPR detection technique, improving microwave transmission doesn't always lead to higher EPR signals. For e.g., the SNR can decrease if the EPR line is oversaturated with too much microwave power. I suggest modifying the sentence to 'more efficient EPR excitation'

Line 34: Can you elaborate on why corrugated waveguides are challenging to be used in the dissolution DNP polarizer system? I do not see obvious reasons why it can't be done. Moreover, the < 0.1 dB/m attenuation (doi: 10.1109/irmmw-THz.2011.6105103) might worth the effort in some cases.

Line 40-45: Another possible microwave transmission strategy is using the quasi-optic setup, which is adopted by Songi Han's group at UCSB and Graham Smith's / Steve Hill's W-band HiPER EPR setup. The quasi-optics setup obviates the use of waveguides, and,

hence, less heat load. The authors might want to comment (and cite) if this is a viable option (or not).

Line 66-69: It baffles me a little about the choice of ordering the two waveguides in your setup. Despite 304 stainless steel is austenitic, and, hence, non-magnetic by definition, it becomes mildly magnetic (more significant than 316) after Industrial processing (mechanical) and thermal cycles. (doi: 10.1016/j.pnmrs.2017.06.002 and doi: 10.1016/j.jmr.2009.03.003). Nevertheless, I wonder if it is not better to swap the order of the waveguide (assuming that the thermal insulation is still good). It is perhaps worth mentioning this fact in the manuscript.

Line 65 and Figure 1: It might be a good idea to include a schematic diagram of how the waveguides are incorporated in your DNP setup (taper / 39,2 cm x 2, etc). This might improve readability.

Line 134: To improve readability, refer to Fig 6 for EPR and Fig. 7 for the power-dependent curve.

Line 136: Please explain the AMC abbreviation because not everyone knows. I assume it refers to the VDI active-multiplier chains. Also, the AMC microwave power (depending on the model) is not very flat across the bandwidth. It might be worth mentioning this or including the power vs frequency plot (you can get it from the supplier's technical data sheet) in the SI.

Figure 3: I am not sure why Fig. 3 is in black and white. Can you provide a coloured version? And label in the figure where is the pencil, wool, fabric, etc.

Line 147: I assume it is a 'train of saturation pulses' with delays between them. Can you specify the length of the delays?

In the 'Results and Discussion' section, Fig. 5 is perhaps the most important figure in the paper. However, I wonder if it is possible to estimate or measure the thickness of the silver that you managed to electroplate on the waveguide using such a method.

Moreover, it is well known that the electrical conductivity of metals significantly increases at low temperatures. Depending on the metal purity, copper can become 100 times more conductive at dissolution DNP temperatures of ~ 1.5 K (see NIST, doi:10.1063/1.555614). Thus, the skin depth could be 10 times thinner, and you might not need to do so many 'passes' with your plating tools. Similarly, the bench-top measurement performed at room temperatures is definitely relevant, but those attenuation values might not be the same in

DNP conditions. I think it is worth mentioning this in the text.

Fig 6 caption: The last sentence structure, '300 seconds DNP per point' sounds awkward.