

# Reply to reviewers comments for "Automated manufacturing process for sustainable prototyping of NMR transceivers"

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Dear Editor, esteemed reviewers, thank you for the very valuable and meaningful feedback. We tried to implement your feedback to the best of our knowledge. The modifications made in the text are highlighted in blue. To ease your evaluation of the changes made you will also find our responses to each of your comments in the text below.

We thank you for the time invested to help us improve our publication.

5 Kind Regards from all the authors Dario Mager

## Reviewer 1

The manuscript reports on the design, fabrication, and electrical characterization of mm-size solenoidal coils. Results of NMR spectroscopy and imaging experiments at 1 T with the realized solenoidal coils are reported. The original contribution of this work is in the technique used to realize the solenoidal microcoils, called "extrusion printing". With this technique the authors demonstrated the fabrication of 1.5 diameter solenoidal coils having a wire pitch from 300  $\mu\text{m}$  to 750  $\mu\text{m}$ , a wire width of about 60  $\mu\text{m}$ , and a wire thickness of 8  $\mu\text{m}$ . The "printed" material consist of 82.3 wt-% of Ag nanoparticles of 12 nm (a commercial product). The resistivity of the "printed" material is not mentioned, but from previous publications of the same authors it might be only two times worse that bulk Ag, which is the material with the lowest resistivity at room temperature. The low resistivity of the printed material and the "automated manufacturing process" makes this approach potentially interesting for the fabrication of solenoidal coils (and most probably other coil geometries) for NMR applications. As mentioned below, I would appreciated a more detailed discussion of the foreseen advantages and limitations of the proposed technique for the fabrication of NMR coils.

Here my suggestions/remarks:

1. Previous literature for the "scalable" fabrication of solenoidal microcoils should be included and compared with the approach proposed here. For example: Rogers, John A., et al. "Using microcontact printing to fabricate microcoils on capillaries for high resolution proton nuclear magnetic resonance on nanoliter volumes." Applied physics letters 70.18 (1997): 2464-2466.

The idea to make micro-MR coils Olson et al. (1995) in a non-manual way has been around for quite a while, as seen in the following publications Rogers et al. (1997); Mager et al. (2009); Badilita et al. (2010); Fischer et al. (2013). We have added these references in the manuscript in line 68.

2. The measured electrical resistivity of the realized solenoidal microcoil should be specifically mentioned.

We thank the reviewer for this remark. We added the following sentences in the line 217: »Based on microscopic measurements and calculations, the total conductive length of some solenoid coils between both adhesive joints is determined. In combination with the LSM measurements the resistivity of one of three coils with  $N = 5$  and  $p = 0.3$  mm is about  $3.96 \mu\Omega$  cm. This value is obtained from the coil's serial resistance of  $4.17 \Omega$  at  $4.98$  MHz minus the mean value of the copper rod assembly and corresponds to about 2.5 times of the resistivity of bulk silver at  $298$  K (David R. Lide, ed., 2005).«

3. LINE 75: I think that the phrase "since for the same magnitude of current flowing through the coil, the temperature rise will be higher for a coil with larger resistance and hence the noise" should be re-phrased. To increase the noise significantly the temperature should increase dramatically (at  $600$  K the thermal noise would be only 1.4 times larger than at  $300$  K). On the other hand, it is certainly important to reduce the value of the resistance because this affects directly the thermal noise and the RF efficiency ( $B1/\sqrt{\text{power}}$ ).

We agree with the reviewer that the effect of temperature increase on the thermal noise of the coil is negligible. We have rephrased the sentence to make this clear.

4. TABLE 1: The unit of the RF efficiency are, most probably,  $\text{kHz}/\sqrt{\text{W}}$  and not  $\text{kHz}/\text{W}$ . I would express this quantity in both  $\text{kHz}/\sqrt{\text{W}}$  and  $\text{T}/\sqrt{\text{W}}$ .

We apologize for this mistake and have corrected the units and also mentioned the values in  $\text{T}/\sqrt{\text{W}}$

5. TABLE 1: It is not clear to me why in this table the RF efficiency is in the order of  $1 \text{ kHz}/\sqrt{\text{W}}$  whereas later (LINE 205) the RF efficiency is about  $30 \text{ kHz}/\sqrt{\text{Hz}}$ . Maybe the authors refer to a different frequency or to a different circuitry ?

Thanks for pointing out the mistake. It was a calculation error which has now been corrected in Table 1. The difference in the RF-efficiency between the measurement and the simulation could be due to the circuitry. The B-field simulation in COMSOL was done without tuning the coil, but the port impedance was set to  $50 \Omega$ . The reason was to compare the B-field distribution of different coil types. The coil used for spectrum acquisition was tuned and matched.

6. LINE 88: (detail) The field B is given but in the equations 2-4 only the field H is given explicitly.

We thank the reviewer for drawing our attention to this point. We have changed that in the text.

7. In several places in the manuscript the authors use the term "measured" when they probably refers to simulation results (e.g., in the caption of Figure 1, I guess that "measured" should be replaced by "computed").

changed in the line 96,109,114,124 and Table 1's caption.

- 55 8. TABLE 2: The meaning of "...of the electrical circuit" is not very clear. I guess that this refers to the fact that in one case the coil is simulated stand-alone whereas in the other case it is simulated when mounted on the PCB. I guess this explain why the self-resonance frequency values reported in TABLE 1 are not the same as in TABLE 2. I would propose to merge these two table and make more clear the meaning of each quantity. I would add also the values of the inductance and the resistance for each of the 9 coils (since the self-resonance frequency is much larger than the operating frequency of 45  
60 MHz, a series L-R model of the coil should be appropriated). And I would definitely also add the width and thickness of the "wire" used for the simulation of each coil (I guess similar to the values reported at LINE 183 of about 60 um and about 8 um, respectively)

We have now added three columns in table 2, where the self-resonance with and without the coil mounted on the PCB are presented. The values in the brackets represent the measured values. The inductance and the resistance of the coil  
65 when mounted on the PCB are also presented with the measured values.

9. LINE 110: Is the Q-factor measured or computed ?

We have changed it from "measured" to "calculated"

10. LINE 115: (detail) Are the required values of the capacitors for tuning and matching with the other coils somehow "problematic" ? Maybe explain the choice of the (N=5, pitch=0.525 mm) coil in a different way.

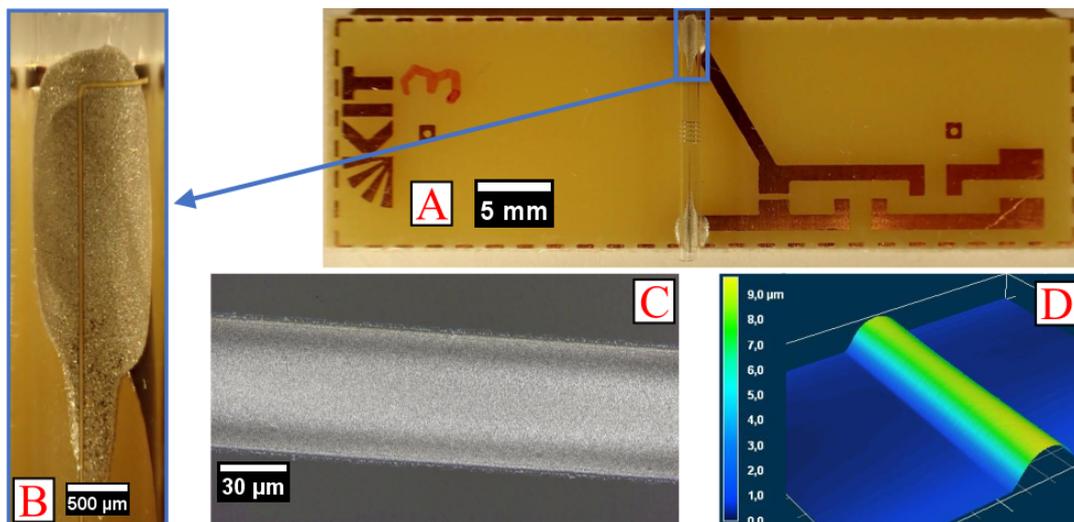
- 70 Tuning any of the other coils is readily feasible if we use trimmer capacitors for tuning and matching. However, as our intention was to avoid non-magnetic trimmer capacitors and to utilize fixed-valued discrete ones, the selected coil with a pitch of 0.525 mm was straightforwardly tunable using discrete fixed-valued capacitors from standard kits. The reason was the large footprint of the trimmer capacitors compared to the coil itself and its cost. We have modified the sentence in the line 130 to clarify this point.

- 75 11. FIGURE 5, CAPTION: (typo) I guess it is "...with p=0.3 mm and N=5 mounted..." instead of "...with p=0.3 mm N=5) mounted...".

Thank you for pointing out the error. We have now fixed it.

12. FIGURE 5: I would add scale bars also in A and B.

We have modified the figure and added the scale bar in A and B.



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13. FIGURE 7: (typo) "Since the Teflon tube show no MRI signal...". (maybe mention that Teflon is based on tetrafluoroethylene ( $C_2F_4$ ) and hence does not contains  $^1H$  nuclei and gives no  $^1H$  NMR signal).

We have changed the caption of the Figure 7 to include the suggestion by the reviewer. The sentence added to the caption is "Since Teflon ( $C_2F_4$ ) has no  $^1H$  nuclei, therefore, it has no  $^1H$  NMR signal. This creates a defined border and highlights the spatial resolution achievable."

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14. LINE 193: Why the "contact resistance" has been measured at 4.98 MHz ? It seems to me an arbitrary frequency not related to the operating NMR frequency, or maybe it is just the same in a very broad frequency range.

We thank the reviewer for this question. The measurements were carried out with an impedance analyser, in the frequency range from 1 MHz to 2 GHz with 801 scanning points. In addition, we measured the mounted coils by means of a simple digital multimeter. For low frequency values within the frequency range used for the measurement, the resistance values measured with the impedance analyzer were similar to the DC resistance values of the multimeter. We decided to use 4.98 MHz, the closest measurement point to 5.0 MHz of each measuring sweep of the impedance analyzer. So, it is an arbitrarily chosen frequency at which the measured values can be assumed as DC measurements, far away from the operating  $^1H$  frequency (45 MHz). We have added a sentence in the line 205 to mention this fact in the paper.

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15. I think that somewhere in the paper the limitations of the proposed printing technique for the fabrication of NMR coils should be discussed more in details. For example: Can the pitch be reduced while keeping the same wire width ? Can the wire width be increased while keeping the same pitch ? Can the wire thickness be increased while keeping the same width ? Which difficulties are foreseen if the solenoidal coil diameter is reduced or increased ? Could we imagine to use this technique for a solenoidal coil having a diameter of 150  $\mu m$  (scaling down also pitch, width, and thickness) ? Is the larger (and positive) magnetic susceptibility of silver with respect to copper a potential problem for high resolution NMR ? Have the 12 nm silver nanoparticles the same susceptibility of bulk silver ?

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We have included the process's limitation in the article's conclusion. In the text we also tried to answer all the questions of the reviewer: The limitations of the extrusion printing process in terms of resolution and minimisation of the line width is mainly determined by the properties of the ink in terms of particle size and rheology, especially regarding its thixotropic behaviour. The linewidth can be reduced by reducing the nozzle's orifice, hence, the distance between the nozzle and the substrate during printing. This distance can be approximated to be 0.5x the inner diameter of the nozzle's orifice. For very small nozzle's orifice, precise automatic control of the distance between the nozzle and substrate is the key. Measuring sub-micron deviations in the stand-off distance when rotating the glass tubes, to be printed on, is challenging and had its limitations with the measurement equipment available when the device was built. Printing linewidths in the range of hundreds of microns was possible but reducing the linewidth further would have led to a reduced conductor's cross-section and hence decreasing the conductivity of the printed tracks. In addition, the tracks need to withstand a certain current. This also limits the miniaturisation of the printed conductor. The pitch of the coils can be reduced to the point where the windings tend to touch when the pitch is equal to the linewidth. For even smaller pitches, a multilayer printing approach is required with additional isolating layers. Vice versa the linewidth can be increased at a constant pitch until the tracks tend to touch. The line thickness strongly depends on the rheology of the ink. Printing parameters and the surface of the glass only have minor influences on the thickness. To increase the thickness of the tracks, the rheology must be adjusted towards higher viscosity and a certain thixotropic behaviour inks. For higher aspect ratios, multi-pass printing instead of single-pass can be a solution, but this can affect the track's edge quality. The main issue when changing the outer diameter of the coil is an appropriate mounting of the tube with the changed diameter. Larger variations in tube diameter due to tube fabrication tolerances probably require a revision of the setup for mounting the tubes. Certainly, a diameter of 150  $\mu\text{m}$  will be challenging and probably not compatible with the bearing blocks of the presented system but with modification of the setup, it may be possible. Since silver ( $-2.31 \times 10^{-5}$ ) Dupree and Ford (1973) and copper ( $-9.63 \times 10^{-6}$ ) Schenck (1996) are diamagnetic, as compared to air ( $3.6 \times 10^{-7}$ ) Schenck (1996), which is paramagnetic, replacing silver with copper did not have any drastic effects. Since the magnetic susceptibility of the silver ink particle is not known, and magnetic susceptibility is a volumetric property, we assume that a bulk of sintered conductive ink should have a similar magnetic susceptibility as bulk silver. However, since the ink particles are small spherical balls, this may have helped to smoothen the effect of susceptibility changes.

## Reviewer 2

The manuscript reports on the the design, manufacturing and validation of a novel additive manufacturing process to produce microcoils for NMR applications. The novelty of the shown approach consists in the specific type of additive manufacturing used that is based on extrusion-printing (EP) of high-viscosity silver nanoparticle-filled ink. The authors designed a range of 1.5 mm ID solenoid coils with varying turn count (3-7) and pitch (0.3 - 0.75 mm) and simulated their electrical properties. The coils were then fabricated using the EP process and characterized in bench-top electrical measurements. A 5-turn / 0.525 mm pitch coil was then used to highlight its properties in NMR-spectroscopy and NMR-imaging experiments.

135 The scientific work is carefully carried out and the claims are supported with the experimental data. Also, the manuscript is well-written and structured. Yet, I propose the following changes to improve the reader's understanding and to better delineate its scope.

1. Please provide an additional paragraph that better delineates the scope and limitations of the novel technique, both in terms of technical limitations (how small can the solenoids get, are there limitations in the pitch/width, etc) and applications (what are the advantages/disadvantages of these new coils compared to the „traditional“ ways, etc)

140 Good point one sometime forgets that the reasons that one found and makes one work over month are naturally not obvious to the reader. We added the following paragraph in the introduction: But why bother in the first place about new fabrication technologies? Conventional solenoidal microcoils can simply be hand-wound Olson et al. (1995) and give excellent results. One reason is that there are other coil designs than just solenoids, i.e. Helmholtz, birdcage, or saddle coils to mention a few Haase et al. (2000). These designs cannot be manufactured manually at the micro-scale. The second reason for direct coil writing is the improved precision. Every material change next to the sample causes disturbances in the magnetic field homogeneity, that is especially a problem for materials that need to be close to the sample like the micro-coil itself. Therefore, it is good to know these disturbances up front so that they can be minimised by design, reducing the magnetic field disturbances that need to be corrected by pre-disturbing the field (so called shimming). Also the electrical tuning and matching gets easier if the coils do show small part to part variations.

145 2. For the readers unfamiliar with additive coil manufacturing, a direct comparison of the main electrical properties to a „traditional“ coil wound from an (enameled) silver or copper wire would be beneficial.

The gain that comes from the high geometrical precision is a little bit reduced by the reduced conductivity of sintered metal tracks compared to bulk wires. The sintered tracks show around 20-70% of the bulk conductivity leading to a slight higher electrical noise. This value was added to the text.

155 3. According to Figs 4/5 the terminals of the coils are connected to the PCB via a long „wire“ running along the capillary. It looks like this section of the conductor is comparable in length to the actual coil conductor:

- Was this part of the simulation (according to Fig 1 it doesn't look like)? And if it wasn't, could this explain some of the reported discrepancies?

160 The electrical lines shown in the figure 4 &5, were not part of the simulation in figure 1. The electrical lines (or the PCB) were simulated by connecting the coil terminal to them as shown in figure 4, separately. The reason for not including them in the first simulation was to get the raw properties of the coil without any electrical circuit connected. The lines were included in the second part of the simulation since this mimics the measuring apparatus with which the coil was characterised. The impedance analyser's probes are at a certain fixed distance, which fixes the geometry of the contact pads of the PCB. With this, we could estimate the effect of the measuring apparatus on the coil.

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- Please describe what the effects of these connection lines are (I expect these to significantly and negatively affect the electrical properties; also the field produced by these sections of the conductor will leak into the lumen of the capillary and might have a negative effect on the spectral and imaging performance of the setup).

170 The connection lines were only part of the electrical characterisation of the coil. The coil was mounted on a different PCB for NMR measurement. The reason for changing the PCB was a space restriction in the magnet. Nevertheless, the coil could be tuned and matched to 45 MHz. If the measurements were to be carried out at higher frequencies, then we would have to be careful as any small electrical path addition would lead to a significant changes in the self-resonance of the coil.

175 4. In various sections of the manuscript it remains somewhat unclear what quantities were simulated and what quantities were actually measured (e.g. „measured“ in figure caption 1 or „measure“ in line 82). Please make sure the wording is clear and consistent throughout the manuscript.

We changed it in the line 96,109,114,124 and Table 1's caption..

180 5. Line 73-75: It seems this argument is incomplete. The primary reason to favor low R's is because it directly affects the thermal noise via  $V_{rms} \propto \sqrt{R}$ . This effect is irrespective of a temperature increase and impacts the SNR even if the temperature of the conductor is constant. A temperature increase due to higher resistance worsens the SNR only as a secondary effect.

We thank the reviewer for mentioning this important point, and we agree that the noise increase due to the temperature increase is a minor effect. We have modified the sentence to clarify this point. Please see, the reply to reviewer 1.

185 6. In the context of the above, the authors could also discuss the relevance of the skin effect:

- For 45 MHz the skin depth of (pure) silver is around 10  $\mu\text{m}$  (and thus about the thickness of the conductor). What is the significance of the skin effect onto the presented results and what does it mean for higher target frequencies?

190 We agree with the reviewer that the skin-depth of the silver-ink at 45 MHz is around 10  $\mu\text{m}$ , and the conductor cross-section used for the measurement was 60  $\mu\text{m}$  by 8  $\mu\text{m}$ . In this case, the current would not decay from one end of the boundary to the other, and in fact will increase the coil's resistance, resulting in the reduction of the Q-factor. However, the effect was not as significant as observed from NMR measurements. For higher frequencies, the skin depth becomes smaller, and as long as the thickness of the conductor is significantly larger than the skin depth, there will be no issue.

- What is the bulk resistance of the cured silver ink compared to pure silver?

195 We thank the reviewer for this remark. We added the following sentences in the line 217: »Based on microscopic measurements and calculations, the total conductive length of some solenoid coils between both adhesive joints is determined. In combination with the LSM measurements the resistivity of one of three coils with  $N = 5$  and  $p = 0.3\text{mm}$  is about 3.96  $\mu\Omega\text{cm}$ . This value is obtained from the coil's serial resistance of 4.17  $\Omega$  at 4.98 MHz

200 minus the mean value of the copper rod assembly and corresponds to about 2.5 times of the resistivity of bulk silver at 298 K (David R. Lide, ed., 2005).«

– What is the surface roughness of the structure and does it impact the skin effect?

Yes, the surface roughness impacts the skin depth of the conductor. The tracks printed had a surface roughness in the range of  $\mu\text{m}$ . Due to the track being printed on the curved glass substrate, it was difficult to determine the exact values.

205 7. Table 1: Please make sure to use the correct units for the RF-efficiency, i.e.  $\text{Hz}/\sqrt{W}$  or  $\text{T}/\sqrt{W}$  instead of  $\text{Hz}/W$  or  $\text{T}/W$  respectively. Does the use of inaccurate units explain the mismatch between the results reported in table 1 vs Line 205?

We apologise for this mistake and have corrected the units and also mentioned the values in  $\text{T}/\sqrt{W}$ . There was also a calculation mistake, which has now been corrected in the table.

210 8. Line 147: I guess the PLC TC1260 TwinCAT 2 software was used to automatically control the manufacturing process after manual adjustment of the substrate placement using the the joystick. Please rephrase.

We thank the reviewer for the suggested change. We rephrased the sentence: »The TwinCAT software is used to automatically control the manufacturing process after manual adjustment of the substrate placement by means of a Logitech F310 Joystick.«

### 215 **Reviewer 3**

The manuscript by Wadhwa et al. reports on a new approach, based metallic paste based extrusion printing (EP), to fabricate micro-coils for NMR and MRI applications. The authors designed, simulated and fabricated a range of 1.5 mm ID solenoidal coils with different number of turns (5, 6, 7) and pitch value (0.3, 0.525, 0.75 mm). Moreover, all coils, connected to a PCB and tuned (matched) to 45 MHz (50 Ohm) are tested using a network analyser for Q-factor assessment. Moreover, driven by  
220 a convenient balance between, RF-efficacy, quality factor and tuning range, the coil with 5 turns and a pitch of 0.525 mm was used for acquiring a a spectrum and an image of glass capillary filled with ethanol.

The paper is well written and conclusions are well supported by results. I only have some minor comments:

1. for non expert readers, it would be nice in the introduction to add a paragraph about why traditional methods for micro-coils fabrication fall short. Is it hard to control  $B_1$  homogeneity? There is a paragraph about  $B_0$  homogeneity that in my  
225 opinion is not the most important factor when the sample volume is small

We thank the reviewer for the questions. We have added a paragraph in the introduction, which answers the question about the shortcomings of micro coil fabrication. Controlling the  $B_1$  field depends on the type and design of the micro-coil, however,  $B_0$  homogeneity plays a crucial role during the design phase of the micro-coil and the choice of the material surrounding it. Differences in wire thickness can cause changes to  $B_0$  homogeneity Esmaeilizadshali et al. (2024). Since

230 NMR signal is a statistical combination of the signals from individual positions. Therefore, in macroscopic measurements  
(for 5 mm or 10 mm test tubes), the number of spins which are closer to the boundary of different materials gets averaged  
out with the large number of spins at the centre position of the detector. Unfortunately, this is not the case for micro-coils,  
where the number of spins closer to the different materials is comparable to the number of spins at the centre position of  
the detector. Although active shimming can help correct the artifacts, however, the shim coils are designed for traditional  
235 macrocoil-type systems. A higher magnitude of the same spherical harmonics would be required to correct for small  
changes in the micro volumes.

2. specify what is the aspect ratio of a printed track and why is so important

240 We thank the reviewer for this question. In the present case, the aspect ratio is the ratio of the thickness to the width  
of the printed track. The higher this ratio, the higher the cross section and the higher the conductivity at a constant line  
width. Hence, the aspect ratio plays an important role regarding further miniaturisation of the coils, as it is hard to reduce  
the resistivity of the printed tracks. We added the sentence »The aspect ratio of a printed line describes the quotient  
of layer thickness and width. This ratio should be as high as possible in order to achieve sufficient conductivity when  
miniaturizing the line width.«in the manuscript line number 199 and 200 as well as the obtained value of 0.14 for the  
analysed track of figure 5.

245 3. correct units for RF-efficacy in table 1 (kHz/ $\sqrt{W}$ )

We have corrected the units in the table.

4. would be nice to add a figure with the Q-factor of the different coils (measured and calculated)

We have added this information now in table 2.

5. how many averages were used to acquire the spectrum?

250 The spectrum was acquired with a single scan. We have added the information in the paper in line 229.

3- specify

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