Journal of Visualized Experiments
Hybrid Printing for the Fabrication of Smart Sensors
--Manuscript Draft--

Article Type:	Invited Methods Article - JoVE Produced Video
Manuscript Number:	JoVE58677R3
Full Title:	Hybrid Printing for the Fabrication of Smart Sensors
Keywords:	Additive manufacturing; Metal 3D-Printing; Inkjet-printing; Multilayer printing; Photonic curing; FIB measurements; SEM measurements; Surface characterization; Profilometer measurements
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Additional Information:	
Question	**Response**
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Journal of Visualized Experiments

Klagenfurt, 26.06.2018

Dear Editor and Reviewers,

I wish to submit an invited manuscript entitled "Hybrid Printing for the Fabrication of Smart Sensors" for consideration by the Journal of Visualized Experiments.

In this paper, we report on a fabrication strategy to build cost efficient and smart sensor prototypes based on additively manufactured and foil substrates for multilayer inkjet-printing.

Such substrates can enable and alleviate the fabrication of smart and functional packages, implants or spare parts. To fabricate functional devices, a multilayer structure composed of an insulating layer and a superposed conductive layer is inkjet-printed onto the various substrates. Additionally, also the interconnects are fabricated in an additive manner using, e.g., low temperature curable conductive adhesive.
The printing and respective curing are done using adapted settings for all of the substrates. The characterization is based on microscopic and focused ion beam analyses, as well as profilometer and contact angle measurements of the substrates. Furthermore, microscopic images and four-point-probe resistance- and profilometer measurements are carried out, to gain insight into the quality of the multilayer print and the substrates' surface as well as bulk structures. We show the diversity of 3D-printed substrates, even for those, which are fabricated using the same process. Additionally, the crucial process parameters and steps are elaborated.

Finally, we can also demonstrate the high quality of measurements acquired with the fabricated prototypes, based on a sensor employing a capacitive measurement principle.

Please address all correspondence concerning this manuscript to me at Lisa-Marie.Faller@aau.at. Thank you for your consideration of this manuscript.

Sincerely,

Lisa-Marie Faller

TITLE:

Hybrid Printing for the Fabrication of Smart Sensors

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KEYWORDS:

Additive manufacturing, metal 3D-printing, inkjet-printing, multilayer printing, photonic curing, FIB measurements, SEM measurements, surface characterization, profilometer measurements

SUMMARY:

Here we present a protocol for the fabrication of inkjet-printed multilayer sensor structures on additively manufactured substrates and foil.

ABSTRACT:

A method to combine additively manufactured substrates or foils and multilayer inkjet printing for the fabrication of sensor devices is presented. First, three substrates (acrylate, ceramics, and copper) are prepared. To determine the resulting material properties of these substrates, profilometer, contact angle, scanning electron microscope (SEM), and focused ion beam (FIB) measurements are done. The achievable printing resolution and suitable drop volume for each substrate are, then, found through the drop size tests. Then, layers of insulating and conductive ink are inkjet printed alternately to fabricate the target sensor structures. After each printing step, the respective layers are individually treated by photonic curing. The parameters used for the curing of each layer are adapted depending on the printed ink, as well as on the surface properties of the respective substrate. To confirm the resulting conductivity and to determine the quality of the printed surface, four-point probe and profilometer measurements are done. Finally, a measurement set-up and results achieved by such an all-printed sensor system are shown to demonstrate the achievable quality.

INTRODUCTION:

Additive manufacturing (AM) is standardized as a process where materials are joined to make objects from 3D model data. This is usually done layer upon layer and, thus, contrasts with subtractive manufacturing technologies, such as semiconductor fabrication. Synonyms include 3D-printing, additive fabrication, additive process, additive techniques, additive layer manufacturing, layer manufacturing, and freeform fabrication. These synonyms are reproduced from the standardization by the American Society of Testing and Materials (ASTM)[1] to provide a unique definition. In the literature, 3D-printing is referred to as the process where thickness of the printed objects is in the range of centimeters to even meters[2].

More common processes, such as stereolithography[3], enable the printing of polymers, but the 3D-printing of metal is also already commercially available. The AM of metals is employed in manifold areas, such as for the automotive, aerospace[4], and medical[5] sectors. An advantage for aerospace structures is the possibility to print lighter devices through simple structural changes (*e.g.*, by using a honeycomb design). Consequently, materials with, for instance, greater mechanical strength, that would otherwise add a significant amount of weight (*e.g.*, titanium instead of aluminum)[6], can be employed.

While the 3D-printing of polymers is already well established, metal 3D-printing is still a vibrant research topic, and a variety of processes have been developed for the 3D-printing of metal structures. Basically, the available methods can be combined into four groups[7,8], namely 1) using a laser or electron beam for cladding in a wire-fed process, 2) sintering systems using a laser or electron beam, 3) selectively melting powder using a laser or electron beam (powder bed fusion), and 4) a binder jetting process where, commonly, an inkjet print head moves over a powder substrate and dispenses binding agent.

Depending on the process, the respective manufactured samples will exhibit different surface and structural properties[7]. These different properties will have to be considered in further efforts to further functionalize the printed parts (*e.g.*, by fabricating sensors on their surfaces).

In contrast to 3D-printing, the printing processes to achieve such a functionalization (*e.g.*, screen and inkjet printing) cover only limited object heights from less than 100 nm[9] up to a few micrometers and are, thus, often also referred to as 2.5D-printing. Alternatively, laser-based solutions for high-resolution patterning have also been proposed[10,11]. A comprehensive review of the printing processes, the thermally dependent melt temperature of nanoparticles, and the applications is given by Ko[12].

Although screen printing is well established in the literature[13,14], inkjet printing provides an improved upscaling ability, together with an increased resolution for the printing of smaller feature sizes. Besides that, it is a digital, noncontact printing method enabling the flexible deposition of functional materials on three-dimensional. Consequently, our work is focused on inkjet printing.

Inkjet printing technology has already been employed in the fabrication of metal (silver, gold, platinum, *etc.*) sensing electrodes. Application areas include temperature measurement[15,16], pressure and strain sensing[17-19], and biosensing[20,21], as well as gas or vapor analysis[22-24]. The curing of such printed structures with limited height extension can be done using various techniques, based on thermal[25], microwave[26], electrical[27], laser[28], and photonic[29] principles.

Photonic curing for inkjet-printed structures allows researchers to use high-energy, curable, conductive inks on substrates with a low-temperature resistance. Exploiting this circumstance, the combination of 2.5D- and 3D-printing processes can be employed to fabricate highly flexible prototypes in the area of smart packaging[30-32] and smart sensing.

The conductivity of 3D-printed metal substrates is of interest to the aerospace sector, as well as for the medical sector. It does not just improve the mechanical stability of certain parts but is beneficial in near-field as well as capacitive sensing. A 3D-printed metal housing provides additional shielding/guarding of the sensor's front-end since it can be electrically connected.

The aim is to fabricate devices using AM technology. These devices should provide a sufficiently high resolution in the measurement they are employed for (often at micro- or nanoscale) and, at the same time, they should fulfill high standards regarding reliability and quality.

It has been shown that AM technology presents the user with enough flexibility to fabricate optimized designs[33,34] which improve the overall measurement quality that can be achieved. Additionally, the combination of polymers and single-layer inkjet printing has been presented in previous research[35-38].

In this work, available studies are extended, and a review about the physical properties of AM substrates, with a focus on metals, and their compatibility with multilayer inkjet printing and photonic curing is provided. An exemplary multilayer coil design is provided in **Supplementary Figure 1**. The results are used for providing strategies for the inkjet printing of multilayer sensor structures on AM metal substrates.

PROTOCOL:

CAUTION: Before using the considered inks and adhesives, please consult the relevant Material Safety Data Sheets (MSDS). The employed nanoparticle ink and adhesives may be toxic or carcinogenic, dependent on the filler. Please use all appropriate safety practices when performing inkjet printing or the preparation of samples and make sure to wear appropriate personal protective equipment (safety glasses, gloves, lab coat, full-length pants, closed-toe shoes).

NOTE: The protocol can be paused after any step except steps 6.3 - 6.6 and steps 9.2 - 9.5.

1. Preparation of 3D-printed Substrates

1.1. Prepare computer-aided design (CAD) drawings, ideally using the stereolithography .stl file format.

NOTE: The used designs are illustrated in **Supplementary Figure 2** and **Supplementary Figure 3**.

1.2. Choose the AM process based on the material properties required by the target application (see **Table 1** for the respective process limitations).

NOTE: In this work, we used samples made of 3D-printed copper, as well as 3D-printed ceramics.

1.3. Fabricate the copper substrate by 3D-printing with wax and lost wax casting[35].

1.4. Fabricate the ceramic substrate by lithography-based ceramic manufacturing (LCM) technology[36] (see **Video 1**).

1.5. Fabricate the acrylate substrate using a high-resolution polymer 3D-printer[37] and remove the supporting wax from the printed part.

1.5.1. Put the printed part inside an oven at 65 °C for 1 h to melt the supporting wax.

1.5.2. After removing the printed part from the oven, put it inside an ultrasonic oil bath at 65 °C to remove the wax from holes, small openings, *etc.*

1.6. Clean the substrates using a wiper wetted with acetone, as possible surface impurities greatly affect the later inkjet print quality.

NOTE: The preparation of AM substrates can be done using different equipment and processes. Depending on the fabrication strategy, the surface and bulk properties may vary as well. It is, therefore, crucial to control these properties using the inspection techniques recommended later (see, for instance, section 4 of this protocol).

2. Fabrication of Interconnects

NOTE: The fabrication of interconnects differs depending on the type (conductive/nonconductive) of substrate.

2.1. Fabricate interconnects on nonconductive (ceramic) substrates.

2.1.1. Dispense the low temperature curable conductive adhesive with a time-pressure microdispenser mounted on a microassembly station into the appropriate vias of the printed parts.

2.1.2. Leave the fabricated interconnects to dry for 10 min at 23 °C and with ambient pressure.

NOTE: For the ceramic substrate, the interconnects can also be fabricated using solder paste and high-temperature curing.

2.2. Fabricate interconnects on conductive substrates.

2.2.1. Dispense the insulating ink all over the vias' (holes/holes in the substrate) circumference *via* a time-pressure microdispenser.

2.2.2. Perform the photonic curing using intense pulsed light as suggested by the ink supplier.

2.2.2.1. Open the tray of the photonic curing equipment containing the substrate table.

2.2.2.2. Move the copper sample to the substrate table of the photonic curing equipment and fix it using the provided magnetic fixtures.

2.2.2.3. Adjust the height of the equipment's substrate table to move the sample to the focus plane of the curing equipment.

2.2.2.4. Close the tray and adjust the curing profile as recommended by the material's supplier for the printed material in the equipment's software interface and press the start button.

2.2.3. Fill the via with low-temperature curing conductive paste (**Table of Materials**).

NOTE: In general, it is possible to use all forms of one-component, epoxy-based conductive adhesives which are temperature activated.

2.2.4. Dry the fabricated interconnects for 10 min at 23 °C.

3. Preparation of the Inkjet Printing System

3.1. Clean/purge the print head nozzles with the purge setting in the printer software, using the appropriate chemical for the respective ink: use isopropanol for insulating inks; use triethylene glycol monomethyl ether for the conductive ink. Purge the nozzles by pressing the purge button in the printer's software interface until the solution ejected from the respective nozzles is clear.

NOTE: The amount of chemical necessary depends on the printer, nozzle, and chemical. In this experiment, approximately 2 mL was used.

3.2. Fill the ink containers with approximately 1.5 mL of nanoparticles silver ink with 50 wt.% metal loading and an average particle size of 110 nm using a syringe, for instance, with a 3 mL barrel, and an 18 G Luer lock dispensing needle.

3.3. Use one print head to jet the ink by pressing the **Start head** button in the printer's software interface.

3.4. Use the pre-adjusted jetting profile of the printer for the jetting of the conductive ink.

3.4.1. Move the print head to the dropview position using the **Go to dropview position** option in the printer software interface and observe the jetting of the ink.

3.4.2. Change the parameters of the voltage profile that is preinstalled for the print head and the print head temperature in order to adjust the drop velocity, shape, and volume. Adjust the ink pressure to avoid any spilling of the ink and to reduce the formation of satellite droplets.

NOTE: For the printing system used in this protocol, the operational maximum jetting voltage was set to 40 V and a jetting profile of 1 µs rise/fall time with 10 - 14 µs hold time was used. The silver ink was jetted at 45 °C. The optimum ink pressure is dependent on the ink level. The voltage in the voltage profile has to be increased or reduced depending on the state (*e.g.*, temperature, viscosity) of the ink and the current temperature of the head, as well as the state of the used print head. To achieve proper jetting, we recommend changing the voltage upward in small steps of 1 V. If there is no improvement in the drop shape, reduce the voltage in small steps of 1 V. Follow this procedure until a stable dropping is achieved.

3.5. Adjust the printing parameters for the insulating ink in the same manner as done for the silver ink.

3.5.1. Use another print head to jet the low-k dielectric material, which is a mixture of acrylate-type monomers.

NOTE: Again, an operational jetting voltage of 40 V and a 1 µs rise/fall time with 8 µs hold time was used in this protocol. The dielectric ink could be jetted at 50 °C. The optimum ink pressure is dependent on the actual ink level. Generally, the used parameters highly depend on the properties of the ink, as well as of the substrate or layer onto which it should be printed. During the fabrication process, the printing parameters might have to be adjusted dynamically. Please refer to the user manual of the printing system on how to properly adjust printer parameters.

4. Inspection of the Surface Properties of the Respective Substrates for Printability and the Adjustment of Printer Parameters for the First Layer

4.1. Perform profilometer measurements to determine the surface roughness.

4.1.1. Put the sample on the substrate table (stage) of the profilometer.

4.1.2. If not homed, home the stage using the home button in the software interface.

4.1.3. Choose the respective resolution and area which is mapped in the software interface.

4.1.4. Place the measurement head at the starting position and start the measurement using the jog option and start button in the software interface.

4.1.5. After the measurement is finished, check the result for consistency (*e.g.*, are the shown heights plausible for the number of printed layers) and save the data.

4.2. Perform SEM inspections as per the user manual to analyze the surface quality.

4.3. Perform contact angle measurements as described in the user manual of the SEM station to determine the wettability properties.

4.4. Fix the substrate on the substrate table using adhesive tape and mark its position appropriately.

4.5. Adjust the nozzle and printing parameters in the settings of the software interface by editing the properties of the print head in the printer's software interface.

4.5.1. Again, move the print head to the dropview position using the **Go to dropview position** option in the printer's software interface and observe the jetting of the ink. If necessary, adjust the printing parameters to optimize the jetting.

4.5.2. Choose a nozzle which ejects well-defined and homogeneous drops of ink for printing.

4.5.3. Enter the number of the chosen nozzle in the printer's preferences.

4.6. Perform the drop size tests to determine the size of one printed drop on the respective substrate.

4.6.1. Print a drop pattern, using a known printer configuration.

4.6.2. Determine the achieved drop size using a calibrated microscope or the inbuilt camera system of the printer.

4.6.3. Ensure that the subsequently used printing resolution is appropriate for the observed ink wetting to fabricate a homogeneous and closed surface (*e.g.*, choose a printing resolution of 900 - 1,000 dpi for a drop size of 40 - 50 µm).

4.7. Perform an FIB analysis (**Table of Materials**), as per the manufacturer's instruction, to ensure a sufficient bulk homogeneity for conductive substrates.

5. Curing Parameter Adjustments for the First Layer

5.1. Print multiple structures, using a layer of the ink used for the first device layer, onto a dummy substrate (*i.e.*, a sample of the same material which can later be disposed and is used for testing purposes only).

5.2. Use thermal curing in an oven at 130 °C for at least 30 min at ambient pressure for the printed conductive silver patterns on a ceramic substrate.

NOTE: Depending on the size of the sample, use a pod to hold the sample inside the oven.

5.3. Use the photonic curing for the insulating ink on the metal substrate.

5.3.1. Open the tray of the photonic curing equipment containing the substrate table.

5.3.2. Move the sample to the substrate table of the photonic curing equipment and fix it accordingly (using, for instance, provided magnetic fixtures).

5.3.3. Adjust the height of the equipment's substrate table, using the table spindle to move the sample to the focus plane of the curing equipment.

5.3.4. Close the tray and adjust the curing profile as recommended by the supplier for the printed material in the equipment's software interface and press the start button.

5.4. Control the homogeneity of the surface qualitatively using a microscope and quantitatively using a profilometer.

5.4.1. Put the sample on the substrate table (stage) of the profilometer.

5.4.2. If not homed, home the stage using the respective button in the software.

5.4.3. Choose the respective resolution and area which should be mapped.

5.4.4. Place the measurement head at the starting position and start the measurement.

5.4.5. After the measurement is finished, check the result for consistency and save the data.

5.5. Repeat photonic or thermal curing procedures using adopted curing parameters if necessary.

5.5.1. Increase the used photonic energy in small steps of, for instance, 5 V in the software interface of the photonic curing equipment if the achieved resistance is too high. Decrease the used energy if the sample shows signs of burning.

5.6. Adjust the equipment parameters for the curing of the first functional device layer so that a conductivity sufficient for the application at hand is reached, but yet no burning of the printed structure occurs.

6. Inkjet Printing and Curing of the First Device Layer

6.1. Fix the substrate on the substrate table using adhesive tape and mark its position appropriately.

6.2. As the first layer is conductive, for the ceramic and acrylate type substrate, use substrate table heating of 60 °C.

NOTE: The temperature must not exceed a temperature which might affect the respective substrate (*e.g.*, the acrylate tolerates only up to 65 °C). This adjustment can be done in the printer settings.

6.3. Adjust the nozzle and printing parameters in the settings of the software interface.

6.3.1. Move the print head to the dropview position and observe the jetting of the ink.

6.3.2. Choose a nozzle which ejects well-defined and homogeneous drops of ink for printing.

6.3.3. Enter the number of the chosen nozzle in the printer's preferences.

6.4. Adjust the used resolution of the print head to deposit a homogeneous layer of ink according to the previously determined substrate properties: for low-wettability substrates, for instance, a large contact angle and small drop size increase the printing resolution. Lower the resolution for high-wettability substrates.

NOTE: The adjustment of the printing parameters can be done in the printer settings.

6.5. Select the appropriate reference point to print the pattern and store its coordinates.

6.6. Load the respective scalable vector graphic (.svg) file and select an appropriate resolution and size, dependent on the desired pattern and the dimensions of the substrate in the printer software.

6.7. Perform printing. Repeat the printing of one layer of ink until the homogeneity of the print is satisfying.

6.8. Control the homogeneity of the printed layer using a calibrated microscope or using the inbuilt camera system of the printer.

6.8.1. Move the camera of the printer to the print position and observe the quality of the print given in the printer's software interface.

6.9. Cure the first layer using the parameters determined in section 5 of this protocol.

6.9.1. For silver ink on a polymer substrate (acrylate, foil), use a 1 ms pulse at 250 V with a reduced amount of energy (525 mJ/cm^2).

6.9.2. For silver ink on a ceramic substrate, use heat curing in an oven as recommended with the ink (*e.g.*, 130 °C for 30 min).

6.9.3. Cure the printed dielectric ink at 200 V with 1 ms pulses and repeat the pulses 8x at the frequency of 1 Hz.

NOTE: The spectra of the emitted light used in photonic curing is quite broad (ultra-violet–near-infrared [UV-NIR]). Still, the amount of UV light is sufficient to initiate the photopolymerization and cure the insulating layer.

7. Inspection of the Surface Properties of the Respective Substrates for Printability and the Adjustment of Printer Parameters for Subsequent Layers

NOTE: Please refer to the user manuals of the measurement equipment to perform the profilometer measurements and microscopy inspections.

7.1. Perform profilometer measurements to determine the roughness and thickness of the printed layer.

7.1.1. Put the sample on the substrate table of the profilometer.

7.1.2. If not homed, home the stage using the respective button in the software.

7.1.3. Choose the respective resolution and area which needs to be mapped.

7.1.4. Place the measurement head at the starting position and start the measurement.

7.1.5. After the measurement is finished, check the result for consistency and save the data.

7.2. Perform contact angle measurements to determine the wettability properties.

NOTE: Refer to the user manual of the measurement equipment at hand on how to properly perform contact angle measurements.

7.3. Perform drop size tests to determine the size of one printed drop on the respective substrate.

7.3.1. Print a drop pattern using a known printer configuration.

7.3.2. Determine the achieved drop size using a calibrated microscope or the printer's inbuilt inspection system.

7.4. Adjust the used resolution of the print head to achieve a homogeneous layer of ink: for low-wettability substrates, for instance, a large contact angle and small drop size increase the printing resolution. Lower the resolution for high-wettability substrates.

7.5. Control the electrical properties of the first layer: for a conductive first layer, use the four-point probe to determine the achieved conductivity.

7.5.1. Put the sample on the substrate table.

7.5.2. Lower the measurement head onto the conductive track, making sure the probe has good contact with the printed structure, to be analyzed.

7.6. For an insulating first layer, make sure the surface homogenously covers the conductor below. Use a microscope for confirming. Verify the insulating properties using a multimeter.

8. Curing Parameter Adjustments for Subsequent Layers

8.1. Print multiple structures, using a layer of the ink used for the next device layer, onto a dummy substrate with an equivalent previous layer.

8.2. Use only photonic curing for all substrates.

8.3. After curing, control the electrical and structural properties of the printed layer: to determine if the conductivity is sufficient, use a four-point probe measurement.

8.4. Control the homogeneity of the surface qualitatively using a microscope and quantitatively using the profilometer.

8.5. Repeat photonic curing procedures if necessary.

8.6. Adjust the equipment parameters for the curing of the subsequent functional device layer.

9. Inkjet Printing and Curing of Subsequent Device Layers

9.1. Fix the substrate on the substrate table appropriately at the previously marked position.

9.2. Adjust the nozzle and printing parameters as determined from the previous step.

9.3. Select the appropriate reference point to print the pattern and make sure the printed patterns are well-aligned with each other to ensure proper functionality of the device afterward.

9.4. Load the respective .svg file with appropriate resolution and size.

9.5. Perform printing. Repeat the printing of one layer of ink until the homogeneity of the print is satisfying.

9.6. Control the homogeneity of the printed layer under a microscope (here, the inbuilt camera system of the printer is used).

9.7. Use photonic curing only for the curing of this layer. Use the parameters determined beforehand for an insulating layer or a conductive layer on the insulator.

9.8. After curing, control the electrical and structural properties of the printed layer: to determine if the conductivity range of the conductive layer is acceptable, use a multimeter.

REPRESENTATIVE RESULTS:

From the SEM images shown in **Figure 1**, conclusions on the printability on the respective substrates can be drawn. The scale bars are different due to the different ranges of the surface roughness. In **Figure 1a**, the surface of the copper substrate is shown, which is by far the smoothest. **Figure 1c**, on the other hand, shows steel, a substrate which is not usable for inkjet printing due to the high porosity and unstable contact angle (see also **Table 2**). In **Figure 1b**, an SEM image of the bronze substrate is shown, and in **Figure 1d**, the titanium sample surface is illustrated.

In **Figure 2** and **Figure 3**, the results of the profilometer measurements are given. These evaluations are necessary to determine the surface roughness of the respective substrates. The metal substrates with a roughness well above ~1 µm (aluminum, titanium, and steel) are not usable for inkjet printing, as the ink tends to be absorbed due to the high porosity and, therefore, inhibits the fabrication of homogeneous layers and reproducible structures. The alumina-based ceramic substrate has a comparable roughness, but due to the different fabrication process, does not exhibit such high surface porosities and can, thus, be used.

Drop size tests, such as illustrated qualitatively in **Figure 4** and gathered quantitatively in **Table 3**, give the achievable drop size and, thus, also the wettability properties for the respective substrate and ink combination. Substrates where no distinct drops are formed either have too little wettability (this is true for the AM metals with a low surface roughness), or they are too porous (this is true for the AM metals with a high surface roughness [*e.g.*, **Figure 4d**]). In **Figure 4a**, the printing result on bronze is illustrated. **Figure 4b** shows copper, **Figure 4c** shows ceramics, and **Figure 4d** illustrates the steel sample result.

In **Figure 5**, microscopic images of the results after the curing of a conductive layer of 1 mm width on insulating ink are given. Based on these images, the integrity of the prints can be assessed. For the conductive ink on copper (**Figure 5b**), the best result can be achieved; the conductive track on aluminum (**Figure 5a**) is completely destroyed; the conductive tracks printed onto the ceramic substrates (**Figure 5c,d**) are intact, but show delamination. The delamination is due to the weak heat absorption and high reflection of the substrates. Reducing the curing dose on these substrates yields conductive tracks which have improved electrical and structural properties.

To determine the height profiles and surface quality of the printed multilayer structures, height profiles, which are the results of profilometer measurements, are gathered, as given in **Figure 6** and **Figure 7**, using the profilometer. From these height profiles, the surface homogeneity of the conductive tracks (the smoothness of the blue curves) can be determined. Additionally, surfaces which lost their structural integrity (aluminum, titanium) can be identified by the large gradients in their height profiles.

The FIB analyses with copper (**Figure 8a**), bronze (**Figure 8b**), titanium (**Figure 8c**), and brass (**Figure 8d**) are shown to illustrate a sufficient bulk homogeneity of AM metal substrates. The scale bars are different here in order to optimally capture the structural characteristics of the multilayer prints (deficiencies in homogeneity, conductive track, *etc.*). This ensures sufficient electrical conductivity of the substrates so that these can be used for shielding in magnetic and capacitive sensing applications. Results for the achieved sheet resistance using a four-point probe are gathered in **Table 4**. Additionally, a qualitative assessment of the printed layers is possible. The granular structures are formed by cured nanoparticles and the layer below is the insulating ink. In, for instance, **Figure 8b**, we see nonhomogeneities (holes, air inclusions) in the printed layers. These result from outgassing during curing. Outgassing can occur when the cure dose for conductive ink on insulating ink is too high. This effect negatively influences the integrity of the printed structures, and excessive outgassing leads to destruction.

In **Figure 9**, measurements results are shown. These results are gathered using a demonstrator which employs a capacitive sensing principle. The smoothness of the curves illustrates the high achievable quality despite the structural deficiencies that might result from the printing processes.

FIGURE AND TABLE LEGENDS:

Figure 1: SEM images of the metallic substrates. These images show (**a**) copper, (**b**) bronze, (**c**) steel, and (**d**) titanium. They are taken at different magnifications as illustrated by the scale bar in the lower right corner of each image. Based on these images, the surface homogeneity can be assessed. This figure has been modified from Faller *et al.*[39].

Figure 2: Profilometer measurements of metallic and ceramic AM substrates. The roughness values R_a and R_q in nanometers are determined according to ISO 4287. For silver, the values are 689.39 nm and 788.06 nm, respectively; for aluminum, they are 2151.19 nm and 2750.38 nm,

respectively; for alumina-based (Al_2O_3) substrates, they are 1210.47 nm and 1737.6 nm, respectively; for zirconia-based (ZrO_2) substrates, they are 559.97 nm and 681.56 nm. The waviness is the more widely spaced surface texture of the substrate. The waviness is the remaining texture in-homogeneity with the roughness component removed. This figure has been modified from Faller *et al.*[39].

Figure 3: Profilometer measurements of metallic substrates. The R_a and R_q values for the respective substrates are, for brass, 414.2 nm and 494.49 nm, respectively; for titanium, 1099.86 nm and 1448.06 nm, respectively; for copper, 307.63 nm and 358.92 nm, respectively; for steel, 1966.95 nm and 2238.78 nm, respectively. This figure has been modified from Faller *et al.*[39].

Figure 4: Drop size tests for metallic and ceramic substrates. These images show (**a**) bronze, (**b**) copper, (**c**) ZrO_2, and (**d**) steel. Distinct drops measured here are marked (where possible) by arrows in the respective image. The determined drop sizes are gathered in **Table 3**. This figure has been modified from Faller *et al.*[39].

Figure 5: Microscopic images of conductive ink printed onto an insulator and an AM metal substrate after photonic curing. The substrates are (**a**) aluminum, (**b**) copper, (**c**) Al_2O_3, and (**d**) ZrO_2. The width of the conductive structure in each image is w = 1 mm. The integrity of the conductive structure on aluminum is completely destroyed, whereas the structures on copper and Al_2O_3 remain intact. This figure has been modified from Faller *et al.*[39].

Figure 6: Height profiles for the conductive tracks on the insulator for metal substrates, determined using the profilometer. This figure has been modified from Faller *et al.*[39].

Figure 7: Height profiles for the conductive tracks on metal and ceramic substrates, determined using the profilometer. This figure has been modified from Faller *et al.*[39].

Figure 8: FIB images of conductive ink on the insulator and metallic substrates. These images show (**a**) copper, (**b**) bronze, (**c**) titanium, and (**d**) brass. This figure has been modified from Faller *et al.*[39].

Figure 9: Plot of the measurement results from a demonstrator device fabricated following the suggested methodology.

Table 1: 3D-printing processes' limitations and tolerances. This table has been modified from Faller *et al.*[39].

Table 2: Gathered contact angles a_c and their standard deviation σ_a in degrees. This table has been modified from Faller *et al.*[39].

Table 3: Gathered drop diameters d_d in micrometers. This table has been modified from Faller *et al.*[39].

Table 4: Gathered sheet resistances r□ in mΩ/□. Sheet resistances are denoted using a square (□) index meaning ohms per square. This term generally refers to 2D-structures and, thus, also implies that the current flow is along the plane of the sheet. The sheet resistance can be multiplied by the film thickness to give the bulk resistivity. This table has been modified from Faller *et al.*[39].

Video 1: LCM process. This process is used to fabricate the ceramic substrates.

Supplementary Figure 1: Example of a multilayer coil design.

Supplementary Figure 2: Example of computer-aided design (CAD) drawings, used for the 3D-printing of multilayer coil structures.

Supplementary Figure 3: An example of computer-aided design (CAD) drawings, used for the 3D- printing of multi-electrode capacitive sensors.

DISCUSSION:

A way to fabricate multilayer sensor structures on 3D-printed substrates and on foil is demonstrated. AM metal, as well as ceramic and acrylate type and foil substrates are shown to be suitable for multilayer inkjet printing, as the adhesion between the substrate and the different layers is sufficient, as well as the respective conductivity or insulation capability. This could be shown by printing layers of conductive structures on insulating material. Furthermore, the printing and curing processes for all layers was successfully performed without impairing each other.

The fabrication strategies presented in this work are highly sensitive to the interplay of the different materials and surface properties. Consequently, the reproducibility of the performed steps is dependent on the respective manufacturing process. For the preparation of the used AM materials, it needs to be considered that the surface and bulk properties may vary significantly depending on the fabrication method (**Figure 1** and **Table 2**). For the inkjet printing, the proposed parameters have to be carefully adjusted to the used printing system, as well as to the respective inks[40,41,42]. The jettability of different Ag nanoparticle inks may vary significantly, depending on the formulation. This means that the ink's solvents and certain additives determine its specific viscosity, surface tension, and boiling point.

Another point to consider is the agglomeration of solid content when the ink ages or is not stored properly, which can distort the jetting quality. Besides that, the specific set-up of the print head itself is also crucial, especially the dimensions of the nozzle opening. It determines the actual jetting parameters, such as the jetting voltage, waveform, and temperature setpoint, as well as the resulting drop size (**Figure 4** and **Table 3**). During the printing process itself, a heated substrate table might also increase the temperature of the print head because of the spatial proximity, resulting in a change and possible degradation of the printing behavior. Therefore, it is crucial to monitor the print head temperature during processing.

Another factor which might influence the jetting behavior during printing is the ink pressure as it might have to be decreased as the ink level lowers during processing. The fabrication of the interconnects on a conductive substrate is not trivial, as the dispensed insulating layer has to have a sufficient thickness to avoid short circuits, but still needs to leave sufficient space to form the interconnects using conductive solder paste.

Furthermore, the adhesion between the three materials has to be acceptable to form stable vias. During the curing process, the temperature tolerance of the insulating layer needs to be considered as well. Therefore, low-temperature curing solder paste has been employed for the respective interconnects. After printing the functional layers, they need to be cured to yield the desired sheet resistance (**Table 4**). Thermal sintering is an appropriate and effective method for the silver patterns if the substrate or the underlying layer has a sufficiently high-temperature tolerance[43]. This is not the case for the insulating layers, which is why photonic curing is employed (**Figure 5**). During the photonic curing process, a large amount of energy is transferred to the sample. Therefore, it is crucial to ensure that the printed patterns have sufficiently dried before the curing process as, otherwise, the remaining solvents might reach their boiling point and may destroy the printed layers due to liquid expansion and the formation of bubbles (**Figure 8**).

Furthermore, sufficient drying is necessary to create layers of homogenous thickness (**Figure 6** and **Figure 7**). Homogenous thickness is necessary for applications where nanometer measurements based on, for instance, a capacitive principle is employed (**Figure 9**). Here, a uniform distance from the sensing electrode can significantly affect the quality[44].

Overall, it can be stated that the choice of optimal photonic curing parameters for the device layers on an insulator is a crucial factor: if the introduced energy is not sufficient, the conductive ink remains unsintered and the sheet resistance is too high for the devices to be electrically functional; by introducing too much energy, excessive heat will be produced in the film and, consequently, the conductive track is destroyed. The copper substrate yielded the best result in terms of sheet resistance (see **Table 4**) and also in the achieved surface quality and integrity of the printed metal track. This might be due to its surface roughness being the lowest among all considered substrates. The substrate reflectivity could be identified as influencing the photonic curing result significantly. The respective substrate reflectivity has to be considered in the curing in order to achieve an optimized result with respect to the applied photonic curing spectrum and profile. This has to be adapted for individual substrates and ink combinations.

In this work, the suitability of AM substrates and foil for inkjet printing was demonstrated. Additionally, the material properties together with the factors essential to the process were determined. A strategy to fabricate working sensor prototypes on foil and AM metal and polymer substrates was presented. Finally, the achievable measurement quality based on measurements done with a demonstrator system was shown. This approach forms an important contribution to the future electrical functionalization of surfaces, enclosures, and other structures that have had a solely mechanical purpose in the design of numerous devices so far.

ACKNOWLEDGMENTS:

This work has been supported by the COMET K1 ASSIC Austrian Smart Systems Integration Research Center. The COMET-Competence Centers for Excellent Technologies-Program is supported by BMVIT, BMWFW, and the federal provinces of Carinthia and Styria.

DISCLOSURES:

The authors have nothing to disclose.

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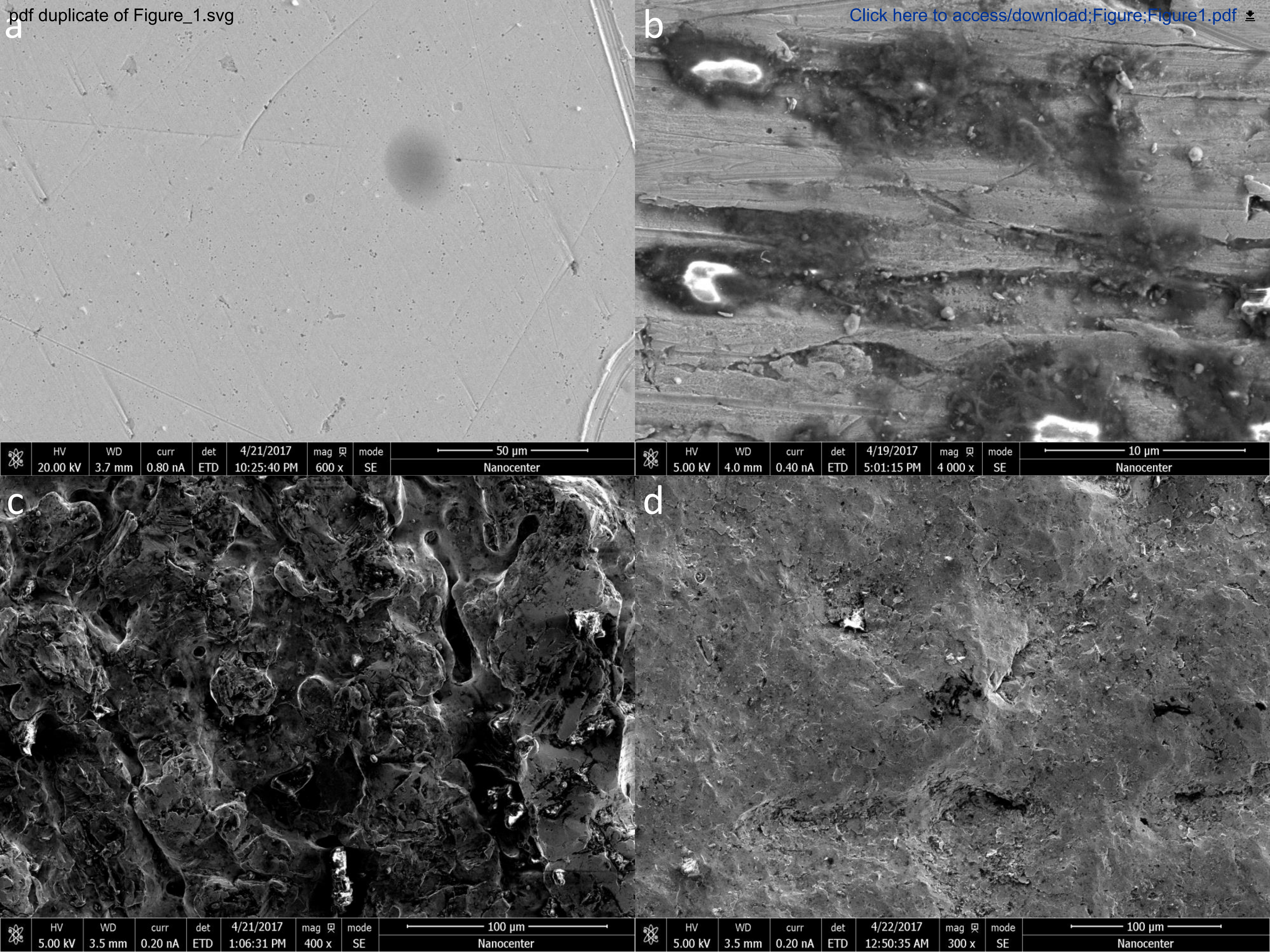
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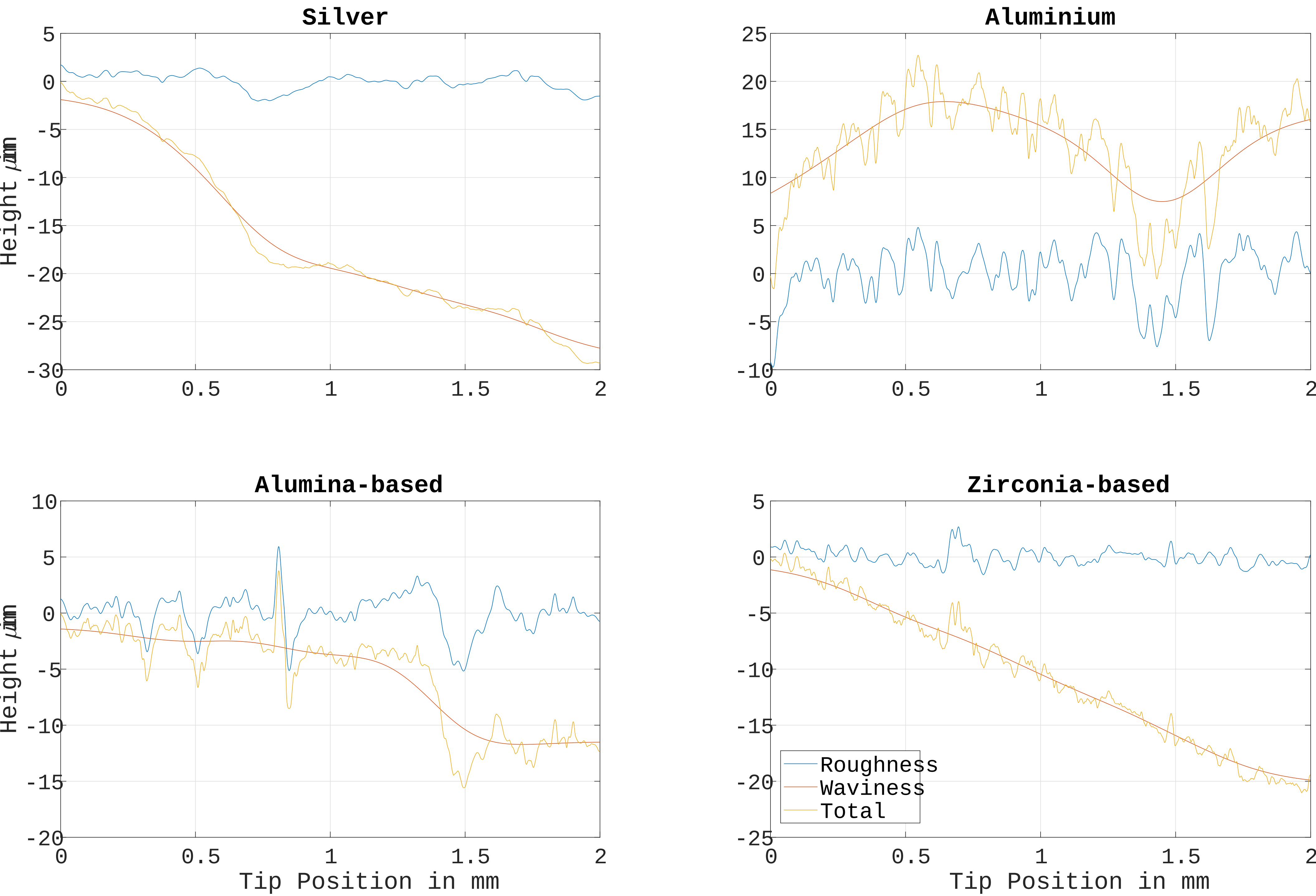

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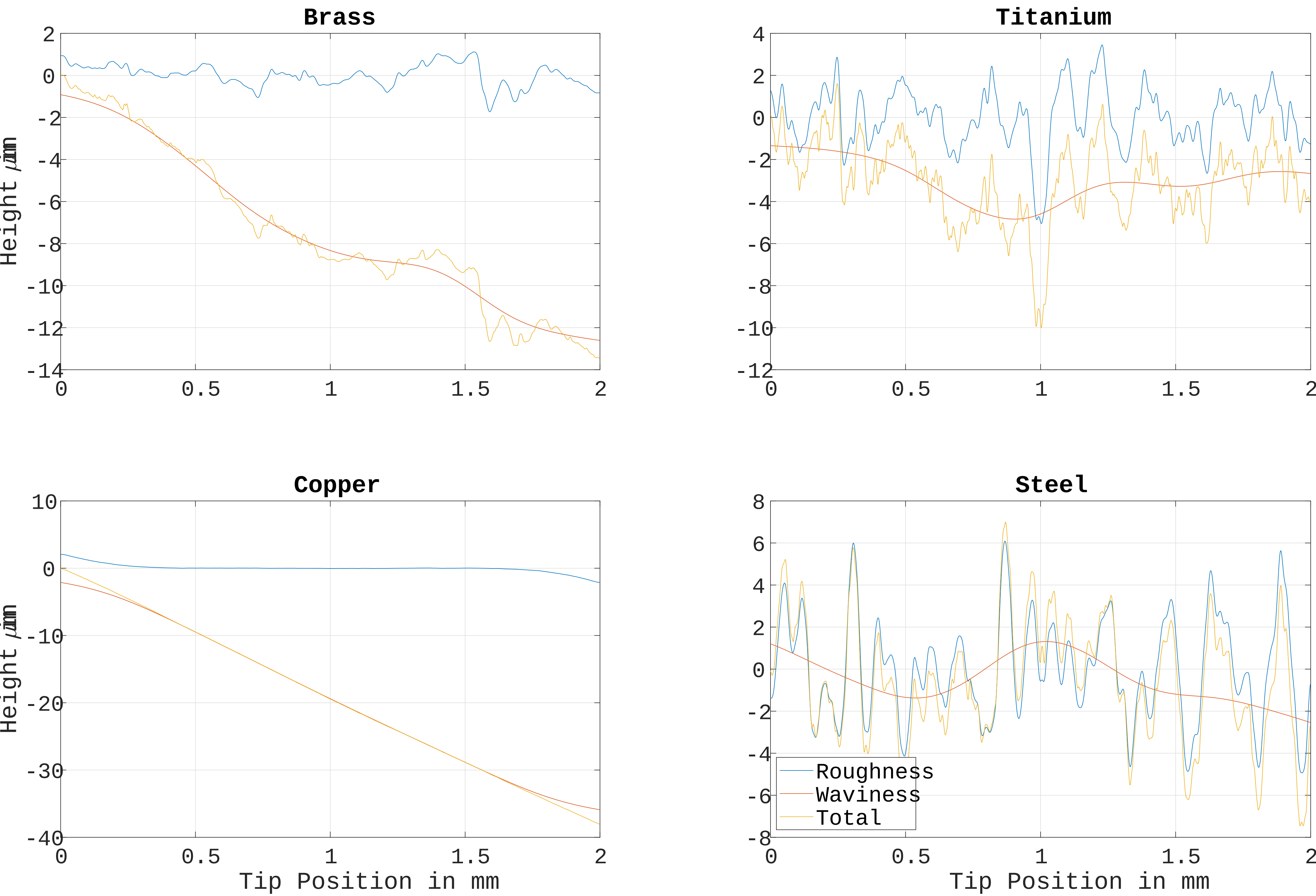

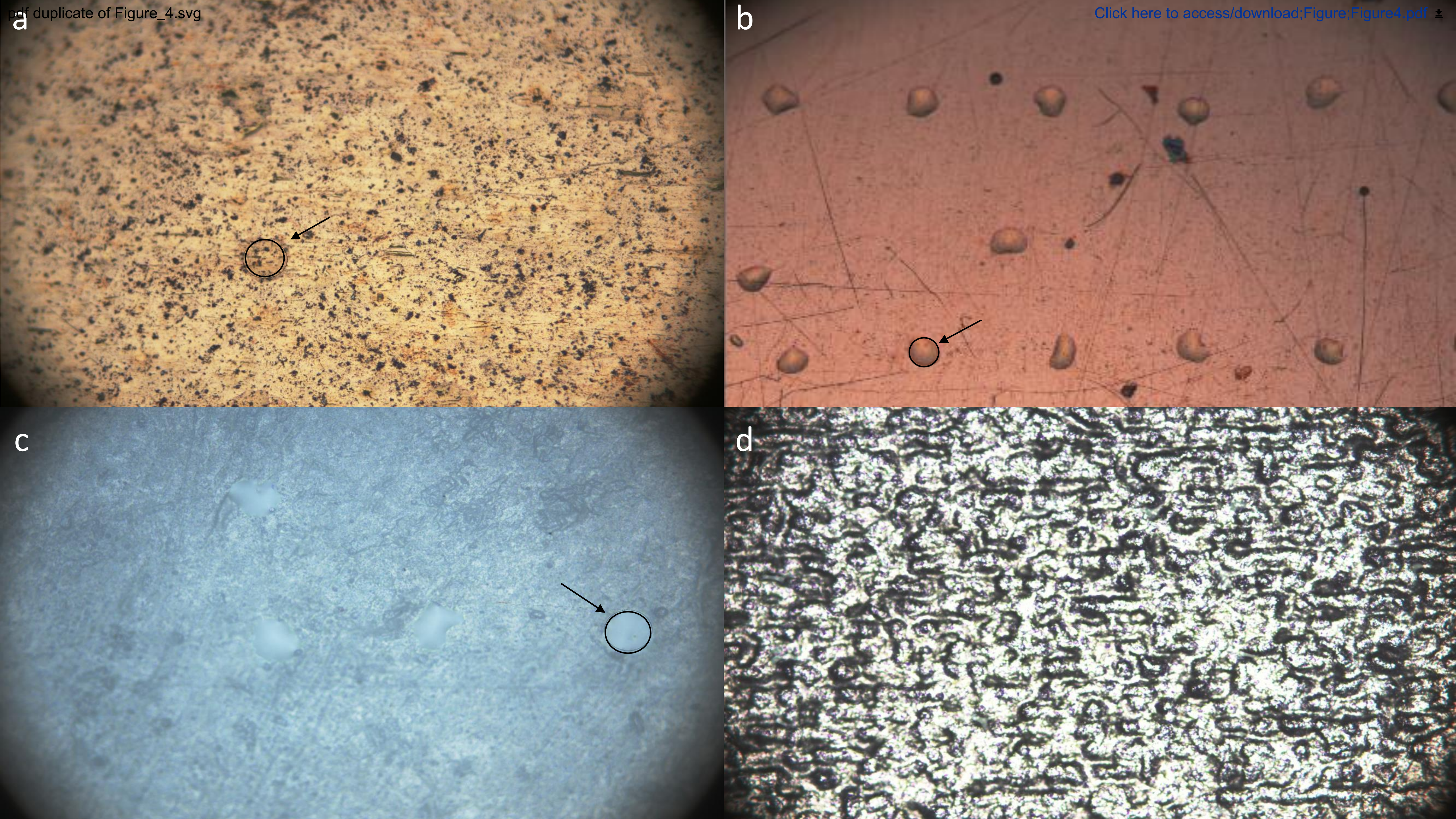
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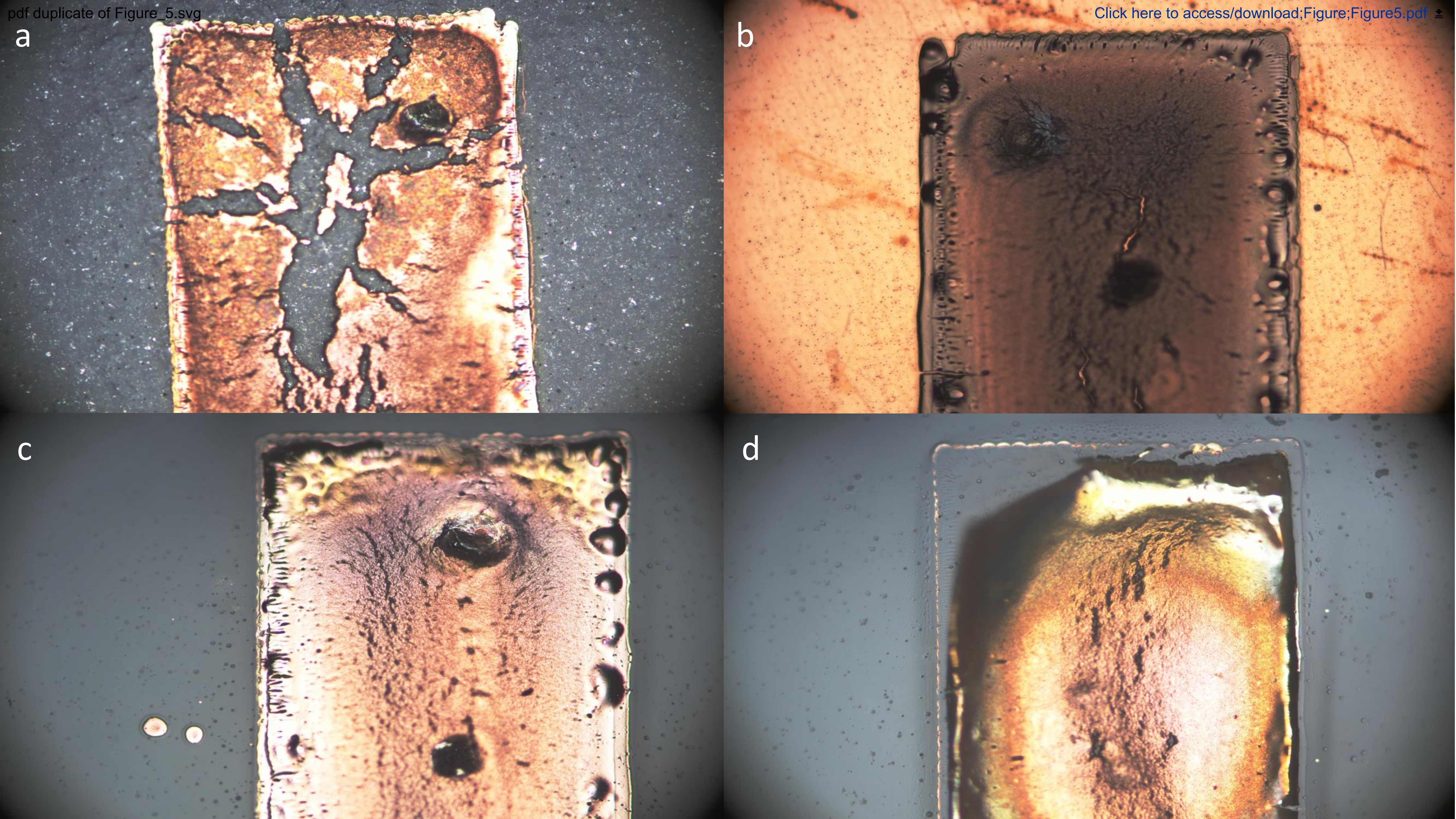
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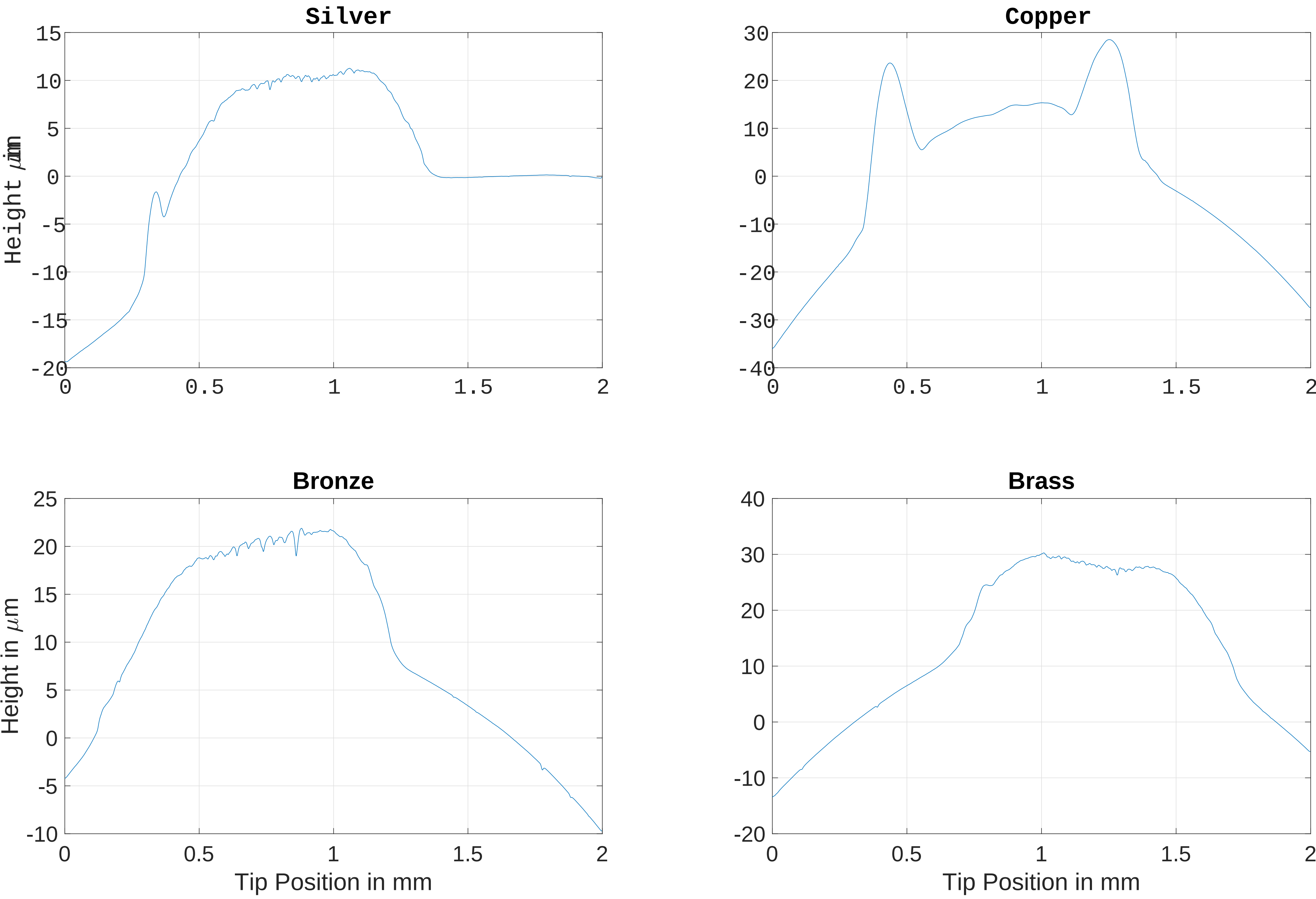

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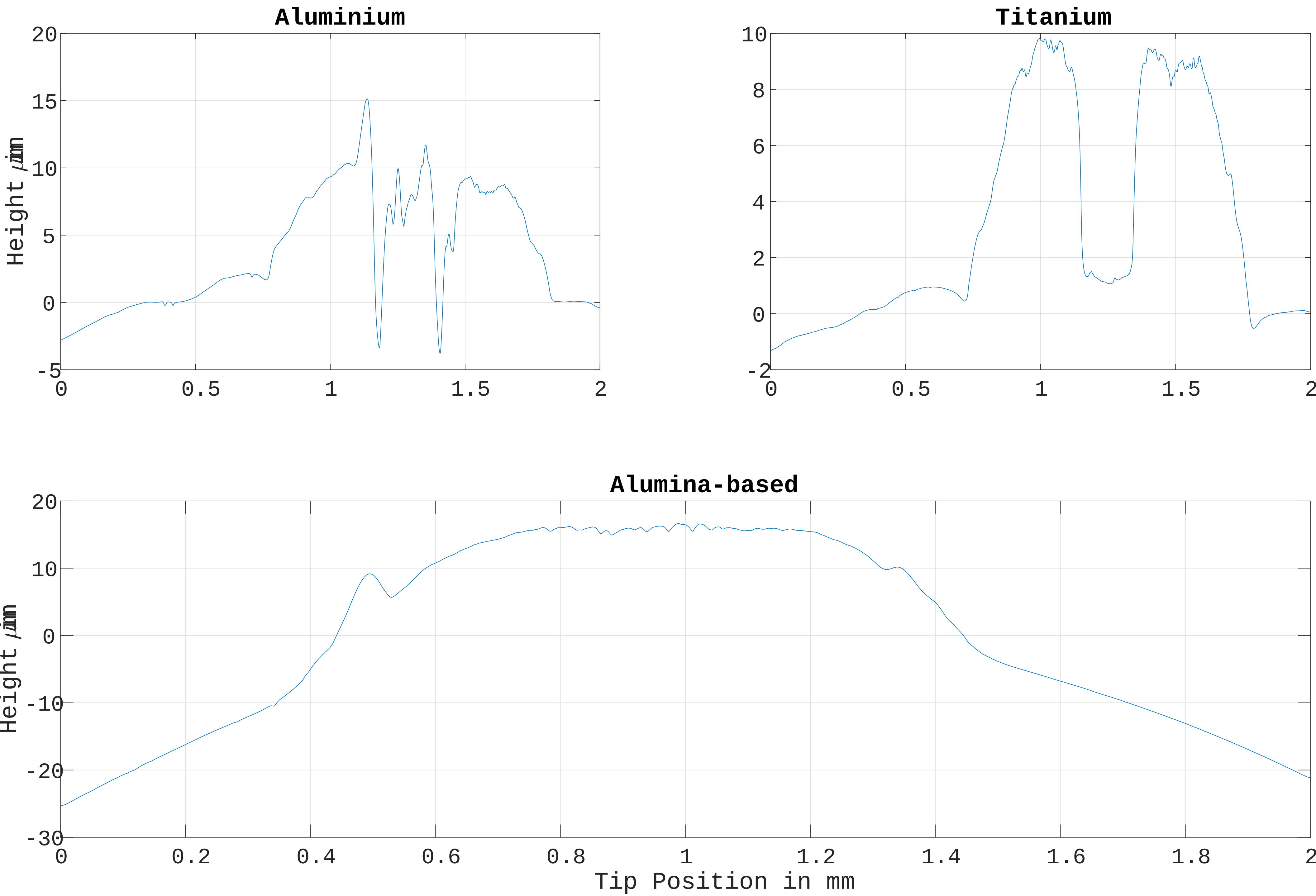
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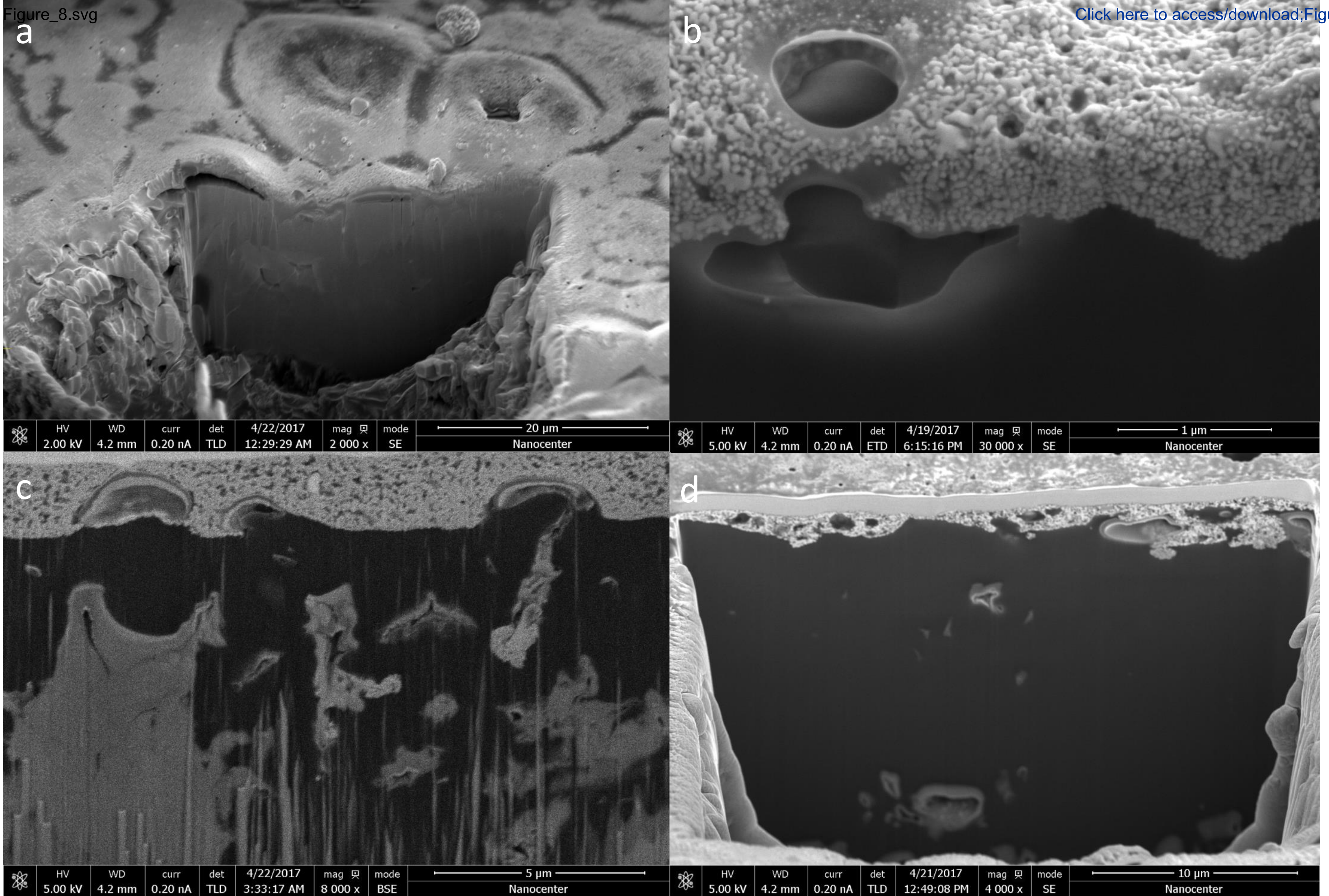
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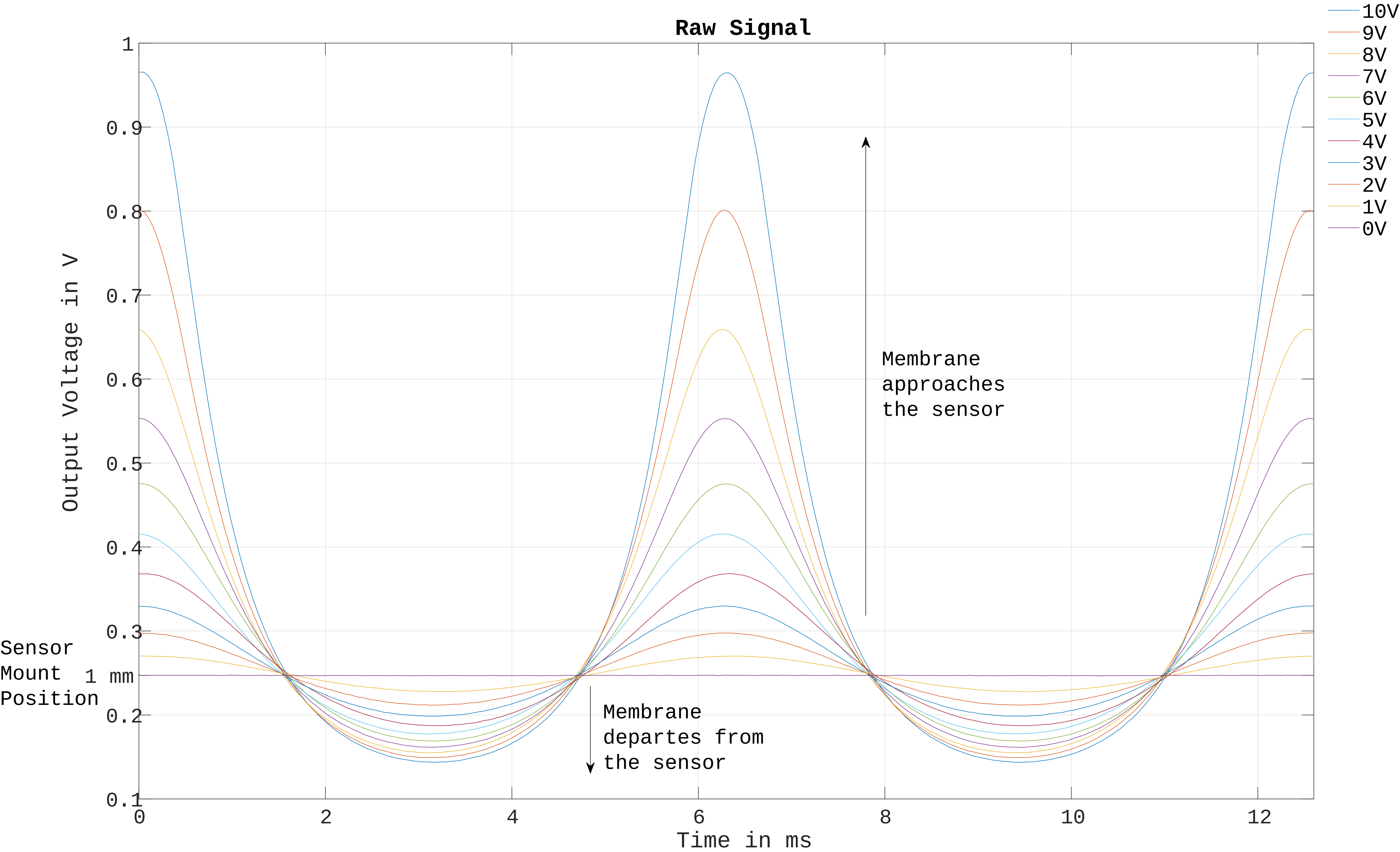

Additive manufacturing process for the ceramic substrates.

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Video or Animated Figure

AM_Ceramics_Lithoz.mp4

SEM images of metallic substrates.

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Profilometer measurements.

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Profilometer measurements.

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Dropsize test results.

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Microscopic images of prints.

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Height profiles.

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Height profiles.

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FIB images of conductive ink on insulator.

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Measurement results gathered with a demonstrator fabricated in the presented methodology.

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Figure_9.svg

	minimum details/ mm	minimum accuracy/ % featuresize	process
Silver	0.25	5.00	wax 3D-printing & lost wax casting
Titanium	0.1	0.2	direct metal laser sintering
Steel	0.35	2 to 3	chemical binding & sintering @ 1300 °*C*
Bronze	0.35	5.00	wax 3D-printing & lost wax casting
Brass	0.35	5.00	wax 3D-printing & lost wax casting
Aluminum	0.25	0.2	direct metal laser sintering
Copper	0.35	5.00	wax 3D-printing & lost wax casting
Al_2O_3	0.025-0.1	0.04	LCM-Technology
ZrO_2	0.025-0.1	0.04	LCM-Technology

Gathered contact angles and standard deviations. Click here to access/download;Table;Table2.xlsx

	titanium	steel	bronze	brass	copper
a_c / °	85.9	71.15	100.3	100.03	88.54
σ_a	7.27	17.64	3.17	2.25	6.84

Gathered drop diameters.

Click here to access/download;Table;Table3.xlsx

	titanium	bronze	brass	copper	Al_2O_3	ZrO_2
dropsize/ µm	23.97	31.3	36.04	29.03	69	69.3

Gathered sheet resistances.

Click here to access/download;Table;Table4.xlsx

	$r_{□}$ in mΩ/□	Comments
Titanium	3000	
Steel	600	
Bronze	2000	
Brass	300	
Aluminium	30000	
Copper	180	
Al_2O_3	150.00	different energy used for photonic curing: 527 mJ/cm²
ZrO_2	20.00	conductive track ablated

Name of Material/ Equipment	Company	Catalog Number
PiXDRO LP 50	Meyer Burger AG	
SM-128 Spectra S-class	Fujifilm Dimatix	
DMC-11610/DMC-11601	Fujifilm Dimatix	
Sycris I50DM-119	PV Nanocell	
Solsys EMD6200	SunChemical	
Dycotec DM-IN-7002-I	Dycotec	
Dycotec DM-IN-7003C-I	Dycotec	
Dycotec DM-IN-7003-I	Dycotec	
Dycotec DM-IN-7004-I	Dycotec	
Pulseforge 1200	Novacentrix	
DektatkXT	Bruker	
C4S	Cascade Microtech	
2000	Keithley	
Helios NanoLab600i	FEI	
SeeSystem	Advex Instruments	
Projet 3500 HDMax	3D Systems	
Polytec PU 1000	Polytec PT	
Microdispenser	Musashi	
Micro-assembly station	Finetech	

Comments/Description

Inkjet-Printer with dual-head assembly.
Printheads with nozzle diameter of 50 µm, 50 pL calibrated dropsize and 800 dpi maximum resolution.
Disposable printheads with nozzle diameter 21.5 µm, 1 or 10 pL calibrated dropsize
Conductive silver nanoparticle ink with 50 wt.% silver loading, with an average particle size of 120 nm, in triethylene glycol monomethyl ether.
Insulating, low-k dielectric ink which is a mixture of acrylate-type monomers. Viscosity is 7-9 cps.
UV curable insulator, Surface Tension: 37.4 mN/m
UV curable insulator, Surface Tension: 29.7 mN/m
UV curable insulator, Surface Tension: 31.4 mN/m
UV curable insulator, Surface Tension: 27.9 mN/m
Photonic curing/sintering equipment.
Stylus Profiler with stylus tip of 12.5 µm diameter and constant force of 4 mg.
Four-point-probe measurement head.
Multimeter to evaluate the measurements using the four-point-probe.
Focused Ion Beam analysis station which provides high-energy gallium ion milling.
Water contact angle measurement device.
Professional high-resolution polymer 3D-printer. See also (accessed Sep. 2018):
https://www.3dsystems.com/sites/default/files/projet_3500_plastic_0115_usen_web.pdf
Electrically conductive adhesive based on Polyurethane, available
Needle for microdispensing.
Equipment for assembly of, e.g., printed circuit boards (PCBs) and placing of chemicals (e.g. solder) and SMD parts.

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Title of Article: Hybrid Printing for the Fabrication of Smart Sensors

Author(s): Lisa-Marie Faller, Matic Krivec, Johanna Zikulnig, Ali Roshanghias, Anze Abram and Hubert Zangl

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Journal of Visualized Experiments

Klagenfurt, 18.09.2018

Dear Editor and Reviewers,

Thank you very much for your valuable comments, we revised the manuscript as requested. In the following, the respective points are addressed invidually.

Editorial Comments:

1. The manuscript was proof-read by three individuals and their suggestions have been incorporated in the text.
2. Lines 41-44 have been rephrased.
3. The copyright permission has been obtained and is uploaded.
4. Unnecessary legends and titles have been removed (the remaining are necessary to identify the measurements belonging to the respective substrates).
5. Waviness was added in the legend.
6. Color arrows have been added.
7. Spaces have been included.
8. Tables have been separated.
9. Table 4 has been filled with the respective data and uploaded.
10. E-mail addresses were added.
11. The appropriate abbreviations were used throughout the text.
12. Spaces were added as recommended.
13. All commercial language was removed from the text.
14. The protocol was revised to contain only action items. In cases where appropriate notes were added.
15. The steps of the protocol were extended as recommended. In few cases, the description of the full procedure would lengthen the text unnecessarily so that a note was added which refers the reader to the user manual of the respective equipment.
16. – 27. All of the details were added in the text and the respective substeps were added accordingly.
28. -30. 2.75 pages of the protocol were highlighted using complete sentences and subclauses.
31. Parts of the discussion Section were moved to the Section on representative results and adopted.
32. References were corrected.

33. Supplementary files were referenced in the text.

Reviewer #1:

1. The Introduction part has been modified accordingly.
2. Regarding the state of the art: there is no comprehensive study which reviews the combination of inkjet-printing on 3D-printed metal substrates. Literature was added presenting further state-of-the-art in terms of metal 3D-printing.
3. Our work presents a study on the combination of 3D-printed substrates, and especially metal substrates, and multilayer inkjet-printing.
4. A paragraph was added which states the content of the following Sections.
5. The format of the protocol Section is as required by the publisher and respective Editors, so that this part can't be changed in its structure.
6. We have shown the suitability for multilayer inkjet-printing by fabricating functional, i.e. conductive and structurally intact conductive layers on insulating layers on 3D-printed metal substrate.
7. We did not measure specifically the porosity of our substrates. However, we know from the details of the LCM fabrication process that it yields homogenous surfaces which is further proven through the surface roughness measurements. In contrast, the binder jetting process yields samples which exhibit much higher surface roughness. Additionally, it can be seen from the dropsize tests that none of the used ink remained on the surface of the substrate. We may thus conclude that all of the ink diffused into the substrate.
8. It is known that polymers exhibit weak heat absorption properties, this is the principle on which photonic curing is based: The conductive tracks are heated excessively on the polymer substrate, but the substrate does not suffer due to its weak heat absorption. Depending on the reflectivity of the substrate surface, where darker surface means less reflectivity, we have to adjust the used photonic energy of the curing equipment. For less reflective substrates saw that we need/can use higher energies whereas for more reflective substrates we have to reduce the used energy. The conductive tracks are printed onto an insulator which itself is printed onto the reflective metal substrate. By qualitative inspection, we found that the insulating polymer layer is not negatively affected by the curing, only the metal layer above delaminates. Thus, we conclude that is due to the combination of high reflectivity of the substrate and weak heat absorption of the insulator.
9. During our fabrication processes we found that it is important to let samples dry before performing the photonic curing. If photonic curing is done readily after printing, and the printed tracks are still excessively wet, the solvent will evaporate instantaneously under the high amount of energy introduced (and the resulting heat), leaving deficiencies in the conductive track.
10. We include your review paper published in Manufacturing Review as well as the review on printed sensors on flexible substrates.

Reviewer #2:

Our manuscript was proof-read by three individuals. Besides minor comments regarding style, they had no objections.

As stated in the introduction of the manuscript, in a common understanding additive manufacturing can be considered as all processes where material is added instead of removed (such as is done in semiconductor manufacturing).
In contrast to this, the ASTM standard terms all processes AM which are based on a digital model or drawing of the desired sample.

Reviewer #3:

1. We added a discussion of metal 3D-printing to our introduction.
2. We added elaborations on inkjet-printing of metal nanoparticles and size-dependent melting temperature drop and references to the introduction.
3. We added also laser-based technologies and references to the introduction part.
4. The mentioned steps of the protocol have been properly extended.
5. A dummy substrate is a substrate which is used only for the sake of experimental determination of parameters and is not used for the fabrication of functional parts.
6. The used conductive paste is given in the Table of Materials. It is a polyurethane-based paste which can be stored at room temperature.
7. This terms has been replaced. The fineplacer is an equipment for micro-assembly and dispensing. It can be used to dispense pastes and solders and assemble SMD parts onto printed circuit boards. The commercially available system which is used in this work is given in the Table of Materials.

Thank you for consideration of our manuscript.

Sincerely,

Lisa-Marie Faller

Logo to be included in the video for the additive manufacturing process for the ceramic substrates.

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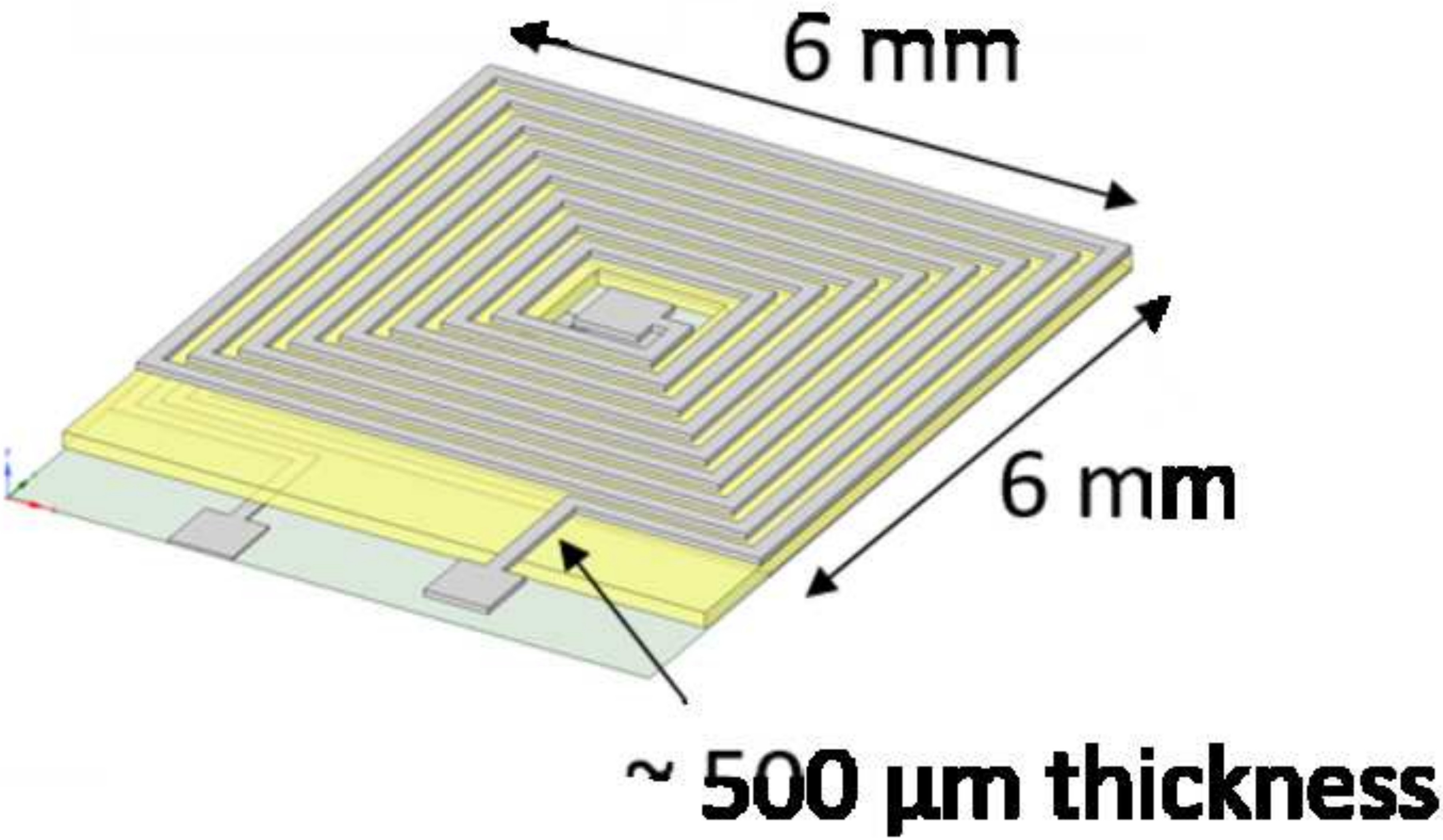
Illustration of a multilayer coil structure as printed onto the respective substrates.



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Licensed Content Author	Lisa-Marie Faller,Matic Krivec,Anze Abram,Hubert Zangl
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Title of the article	Hybrid Printing for the Fabrication of Smart Devices
Publication new article is in	Journal of Visualized Experiments
Publisher of the new article	Other
Author of new article	Lisa-Marie Faller
Expected publication date	Mar 2019
Estimated size of new article (number of pages)	13
Requestor Location	Alpen-Adria-Universität Klagenfurt Universitätsstrasse 65-67

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Publisher Tax ID	GB 494 6272 12
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Drawing of the substrate for a coil structure (inductive) sensor.

Drawing of the substrate for a electrode structure (capacitive) sensor.

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Supplemental Coding Files

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