Final Report

Metadata Framework and Rich Provenance Tracking system for Conductive Inks in Printed Electronics - ReCIPE

1. General Information

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- Project title: Metadata Framework and Rich Provenance Tracking system for Conductive Inks in Printed Electronics – ReCIPE (NFDI4Ing Seed Project)
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2. Executive Summary

ReCIPE addressed the need for a comprehensive description of complex experimental data collections, in particular including metadata on process parameters in printed electronics. While experimental data and methods are commonly reported as "best outcome" figure of merits, the systematic process parameters (while being crucial for achieved outcome, in particular in combination and chains of different process steps) are still often neglected or buried in hard-to-access lab books and similar sources. This complicates and prolongs process optimization, especially when different printing and post-processing steps need to be combined for a specific application. ReCIPE as NFDI4Ing seed project aimed at setting up a tool for researchers and users of printed electronics at the Karlsruhe Institute of Technology (KIT) that can grow into a broad open database on process parameters and connected outcomes for the wider academic and industry based community.
During the course of the project, we identified Kadi4Mat as our platform of choice, established and implemented a data and metadata structure for the use case of conductive metallic inks (Au, Ag, Pt), and started filling the framework with experimental data.

We are now filling the system with additional experimental data, add further processes and reaching out to academic and industry based colleagues to let the system grow into an exhausting provenance tracking for the samples and data collected along different process chains.

3. **Work Report and Results**

Printed electronics is striving to to develop novel applications in various research fields with the help of additive manufacturing processes, and more sustainable techniques, for inexpensive and mass available devices, in particular on flexible substrates e.g. for sensing or biomedical applications. One of the key advantages in printable electronics is the wide range of materials that can be used in many different printing methods. On the other hand, the same diversity in materials and methods bring along the challenge of finding the ideal choice in the combination of material, method and accompanying processing parameters for a given application or target device. This is a widely unmet need of the community as we are struggling to exchange information on methods, process chains and sample treatments as well as the obtained device performances in a standardized and openly accessible manner.

The main aim of ReCIPE was to establish a data and metadata structure for the use case of conductive inks, based on metallic inks, as being a pressing topic, since the conductivity obtained from printable conductive inks after processing often remains behind expectation.[1] The first step of our project was to identify a suitable platform in which we could record and connect metadata on samples, experiments and process steps and the experimental results. For this, we reached out to colleagues and existing initiatives for discussion and screened the available options by looking at existing implementations, and talking to users about their experience with the systems. Quickly, as platforms of interest eLabFTW,[2] Kadi4Mat,[3] and LabIMotion/Chemotion[4] stood out, as all of them satisfied our basic wishes of selecting an open access, open source and free to use tool. After intensive research into the details and capabilities of each platform, we chose Kadi4Mat as our platform of choice for several reasons: (i) it offered the best balance of existing structures and freedom of modelling our own processes, and seemed the most advanced project in regard to features important to the printed electronics community. (ii) As in-house development at KIT, we had direct contact with the developing team for frequent discussions allowing for swift implementation of suggestions for improvement or fixing of software bugs. (iii) It has already gathered a critical mass of users, which gives us trust of continuous growth and support in the future. (iv) The existing users and additional new joining users of Kadi4Mat during the project runtime allowed for additional synergies in particular with the ELN initiatives currently evolving in the Karlsruhe Nano Micro Facility (KNMFi).[5]

After this, a dedicated position was installed (MSc. Mohammad jan Nazami) for the interview phase with members of the different groups in the research unit Aghassi and neighboring groups working on printed electronics, to survey the details of process chains and machines involved in them, as well as what parameters are typically recorded and what experimental data is derived in the end. This data was then modelled into actual process descriptions and implemented into templates and flexible process chains. This process was constantly supported by the proposers and additional personal within the Research Unit (RU) Aghassi (namely Dr. Gabriel Cadilha Marques, Dr. Navid Hussain, and MSc Srivatsan K. Vasantham). Additionally, regular meetings with Dr. Michael Seltzer (lead of Kadi4Mat development at KIT) and one of his staff (Dr. Rihab Al-Salman) were installed and were extremely helpful in implementing the developed ontology and modelling of the identified process chains into the Kadi4Mat platform. The fruitful discussions also lead to several improvements in both directions, our approaches to implementations as well as well usability and functionality of the Kadi4Mat platform.

As result of our efforts, we successfully could implement a model process for printing of metallic nanoparticle inks that was filled with pilot experiments (Figure 1).

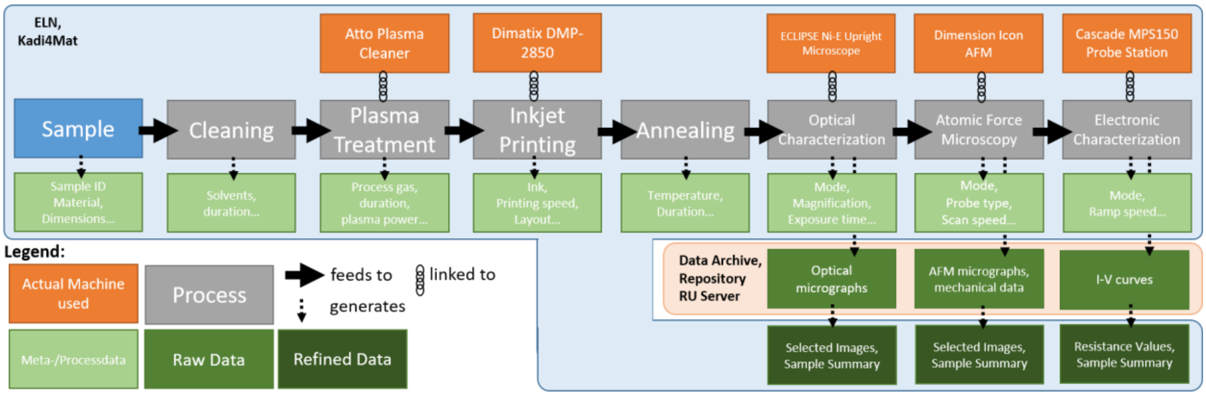

Figure 1. Model process of printing conductive test patterns with inkjet printing, characterizing resulting structure morphology, measuring their conductivity and recording all the relevant metadata along the lifetime of a specific sample. While defining the generic process steps (Plasma Treatment, Inkjet printing, Optical Characterization, …) the user can link the actual instrument or machine used from the instrument database. This then also attaches pre-defined templates of the meta- and process data a specific machine will generate so that the user does not forget to record these. While meta- and process data is stored directly in Kadi4Mat, the full raw data is places into a data archive with adequate storage capacity and linked from the Kadi4Mat experiment record. User selected refined data and summaries are stored directly in the Kadi4Mat experiment, to enable an easy access of end results for collaborators and colleagues or sharing to external researchers.

The framework was implemented by generating various templates for samples (recording mandatory fields as sample IDs, responsible scientist, material, dimensions, time stamps, and so on, as well as optional additional information e.g. on special precautions for handling or safety concerns), processes (e.g. substrate cleaning, pre-treatmeants like silanization, coating, post-treatments like annealing) and the printing process technology itself (e.g. inkjet printing, capillary printing), again recording mandatory process parameters relevant to the specific process technology (e.g. writing speeds, environmental parameters, ...). For each process step, instrument records were generated giving information on the specific instrument type used, that can be selected by the user (e.g. which of different available inkjet printer models within the RU was used) to ensure that relevant standard information and parameters are consistently recorded. Similarly, standard records were created for materials, in particular the used nanoparticle inks, containing information on basic ink properties (e.g. formulation, vendor, date of purchase, batch numbers, ...). While these are – in contrast to the instrument records – much more prone to frequent changes and thus we allow to create these also by the users themselves, this approach again improves greatly the usability as well as consistency of the process recording. The choice of not defining (fixed, not user modifiable) complete processes as a template was made, because it turned out, that in light of the overall flexibility in the printing processes (i.e. the combination of pre- and post-treatments, and the interdependencies of the used materials onto the necessary and optional process steps) is so vast, especially when taking into account that in particular the characterization steps include often very different combinations and order of optical, electron and atomic force microscopy in various modes for each, that users will need to adjust these for almost every experiment. User compliance in the correct and consistent use of these platforms will be much higher by maximizing flexibility and usability by allowing them to bring together these atomistic pre-defined templates as they need for a specific process. With the growing number of processes recorded in the platform they can then take a matching process if available or built new ones from scratch. Therefore, special care was taken to design these building blocks in a way that they are not only useful in the process / experiment at hand (i.e. the printing of interconnects with conductive metallic inks), but flexible enough to be reused in future additional processes and experiments (as e.g. printed transistors and memristors as envisioned as the next class to be implemented).

The construction of a typical experiment record with an actual experiment on conductive test patterns from the atomistic templates is shown in Figure 2.

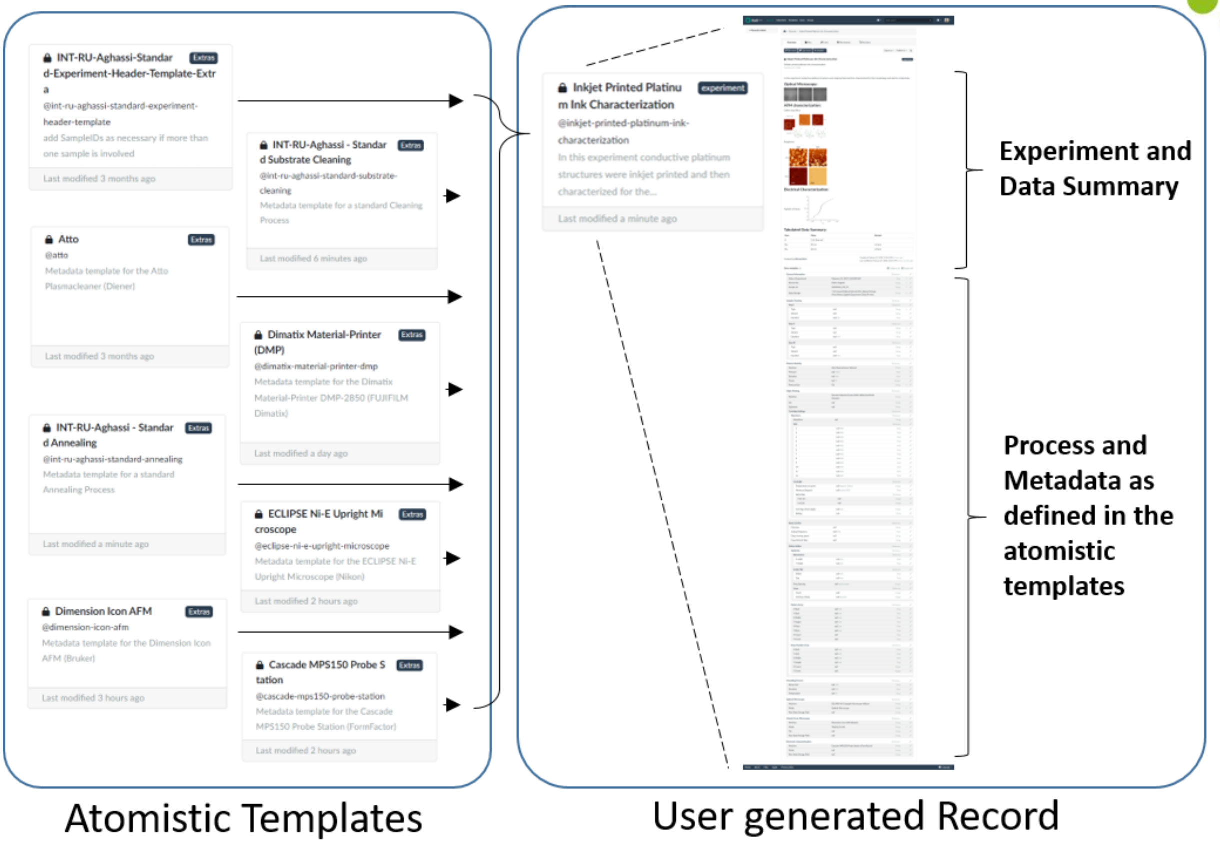

Figure 2. Construction of a typical experiment record. The user selects the standard templates representative for the planned process chain which then built the experiment record for a specific experiment and process chain. Here the user can enter an experiment and data summary presenting selected refined data and end results and notes. Followed is the tabulated meta- and process data that also documents the process chain and links to the location of the raw data. By the unique sample ID, several of such experiment records can also be linked when a sample passes through additional follow up experiments, which assures provenance tracking.

As of writing this report, we have recorded complete data sets of fabrication and characterization for inkjet printing of interconnects with Au, Ag and Pt nanoparticle formulations under different process parameters in regard to morphology and conductivity. This is a strong basis for the envisioned database, as now, additional experiments with an even broader parameter space as well as new ink formulations can quickly be added. Currently, Mr. jan Nazami and an additional PhD student (MSc. Mahsa Saghafi) are using the platform as developed during the project runtime and add in additional sets with more ink formulations and process parameters.

The platform is now already open for use also of colleagues not only in our RU but also to collaborators in the institute and even to outside collaborations (in particular in projects in the framework of the KNMFi). Here we expect great improvements in structured data exchange and sample provenance tracking, as the platform ensures that all necessary information and metadata is reliable available for all participants in a project. After completion of a project, selected data can be made available to the public and interested outside researchers in concordance with FAIR principles and additional raw data storage in open repositories.

An additional synergy already evolved out of the machine records. While we already started to import all machines within the RU Aghassi into the platform (which includes also non-printing related installations), we could leverage Kadi4Mat as the general platform for a future institute-wide machine database (Figure 3), which will allow a seamless integration into processes spanning over users of different groups and RUs. In the future, all KIT researchers and also outside collaborators can add instrumentation and processes into our platform that they used in joint projects. As an added benefit, this machine database can also inform interested persons in the available options in specific labs.

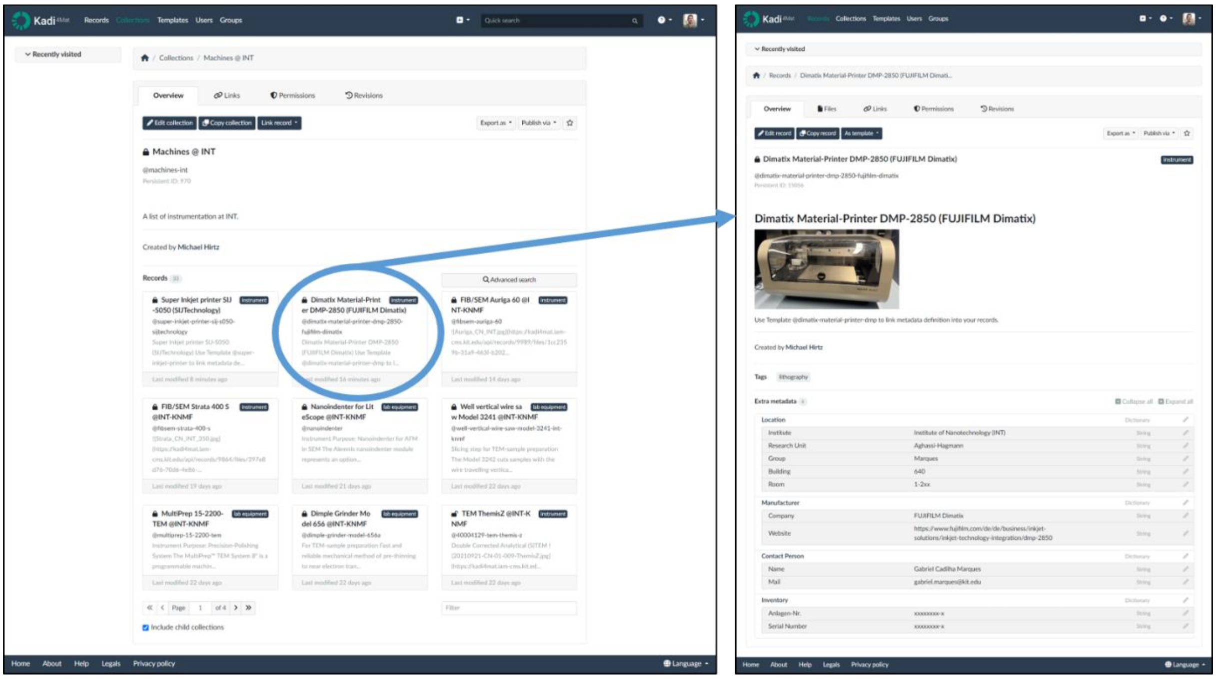

Figure 3. INT wide instrument database. Each instrument has an own record giving basic information on the machine and providing a link to the metadata template that can be incorporated into the users own experiment workflow.

The results of our seed project are subject to a publication in preparation that we plan to submit after a critical mass of experiment sets that are open to the general public was generated. This will then finally disseminate our platform to outside users that can then contribute with additional data and processes to make them accessible in a standardized way useful for developing new or selecting existing process chains in printed electronics. Overall, the seed project ReCIPE did not only allow us to install the basis for a process and result database in regard to metallic interconnect fabrication in printed electronics, but also gave a boost to the development in digital RDM and ELN use in a much broader context.

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Literature and Links:

[1] E. Dimitriou, N. Michailidis, *Nanotechnology* **2021**, *32*, 502009.
[2] "eLabFTW - a free and open source electronic lab notebook," can be found under https://www.elabftw.net/, **2023**.
[3] "Karlsruhe Data Infrastructure for Materials Science," can be found under https://kadi4mat.iam-cms.kit.edu/, **2023**.
[4] "The LabIMotion Extension," can be found under https://chemotion.net/docs/labimotion/, **2023**.
[5] "The Karlsruhe Nano Micro Facility (KNMFi)," can be found under https://www.knmf.kit.edu/, **2023**.