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Contact: Prof. Bassam Z. Shakhashiri

UW-Madison Department of Chemistry

bassam@chem.wisc.edu

www.scifun.org

Polymer Resins with Enhanced Functionality

By

Julia M. Fraser

A dissertation submitted in partial fulfillment of
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The dissertation is approved by the following members of the Final Oral Committee:

Andrew J. Boydston, Professor, Chemistry
David M. Lynn, Professor, Chemical and Biological Engineering
Whitney S. Loo, Professor, Chemical and Biological Engineering
John F. Berry, Professor, Chemistry

6 Summary of My Thesis for the General Public

6.1 Importance of Science Literacy

I noticed that growing up, math and science skills were perceived as natural or “you either have it or you don’t.” When someone said, “I’m not good at math,” the response was often, “It’s okay. Neither am I.” Being bad at math was okay. I have even heard adults make similar statements. But if the same was said about reading, we would not be okay with kids (or adults) being bad at reading. Certainly, we all have natural talents for some skills and difficulties with others, but I think it is important to have a base level knowledge about a variety of subjects, and thus, expend a base level of effort learning about them. History is not my forte. A couple years ago, I had a nightmare that my middle school called and told me I failed eighth grade history and had to go back and take it again. On any given day, I probably could not tell you what year Columbus sailed the ocean blue, but I still try to understand the highlights of history that influenced today.

The biggest challenge to understanding science is the technical vocabulary and complex descriptions that often accompany published work. Even I, a soon-to-be PhD, can struggle with deciphering works from scientific journals, especially those further away from my area of expertise. From a writer’s perspective, I understand. It is easy to forget that much of the knowledge that has become ingrained in my mind over the years has not been for others. Sometimes, I fear that explaining a concept in more basic terms will come off as insulting to those who may already understand it. However, simplicity is appreciated. The best communicators adjust to explain concepts to any audience. Science creates new technologies, but it can only get that opportunity if we can convince the entire community, including non-experts, of its importance. This is why I am including a chapter written for a general audience in my thesis.

6.2 Types of Chemistry

Broad categories within chemistry include organic, inorganic, analytical, biological, physical, and materials. Organic chemistry is the study of carbon-containing compounds and inorganic chemistry is the study of non-carbon containing compounds. Analytical chemistry focuses on using instrumentation to characterize substances. Biological chemistry focuses on chemistry that occurs within living things. Physical chemistry applies physics principles to chemistry. Although all fields of chemistry require some interdisciplinary work, materials chemistry is perhaps the most interdisciplinary in that its goal is to design new materials and to do so, pulls from all other chemistry subfields as well as disciplines in engineering. There are broadly two categories of materials: “hard” and “soft.” Hard materials, like metals and ceramics, are characterized by rigid, high-density structures that are resistant to deformation. Soft materials, like plastics and gels, are flexible and easily deformed by temperature or force. Hard materials are often inorganic and soft materials are often organic. However, “hard” materials can feel soft and “soft” materials can feel hard.

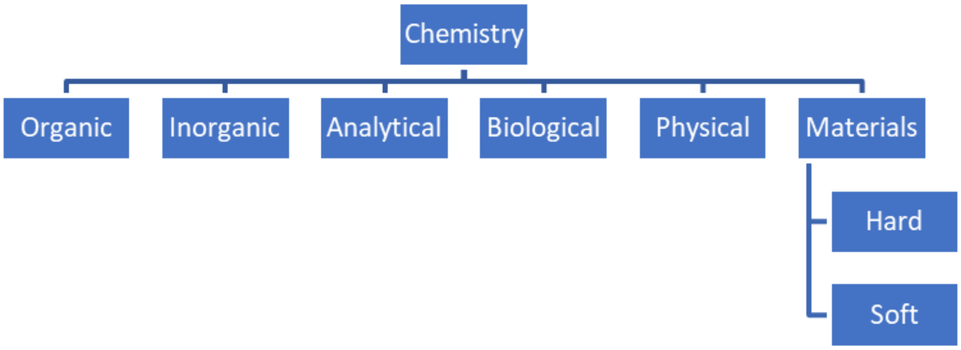

Figure 1. Flow chart of different types of chemistry.

6.3 Defining a “Polymer” and Its Importance

The word “polymer” derives from the Greek words “polus,” meaning “many,” and “meros,” meaning “parts.” A polymer is a chain comprised of many repeating subunits called "monomers." Imagine a monomer is a piece of pipe and a polymer is several pieces of pipe put together. An "initiator" is the inlet, which is where the water comes out. Every pipe needs to start at the inlet just as every polymer needs an initiator to start the process of attaching monomers. There are two types of polymers: "thermoplastics" and "thermosets." A linear string of monomers is called a "thermoplastic." Thermoplastics can melt and can be recycled into their monomers. Examples include disposable water bottles, fuse beads, and Nylon. A pipe fitting, or a joint, that has multiple holes represents a "crosslinker." By connecting more than two monomers with a crosslinker, a polymer can become "crosslinked," forming a "thermoset." Thermosets cannot melt or be recycled into their monomers. Examples include billiard balls, cookware, and silicones.

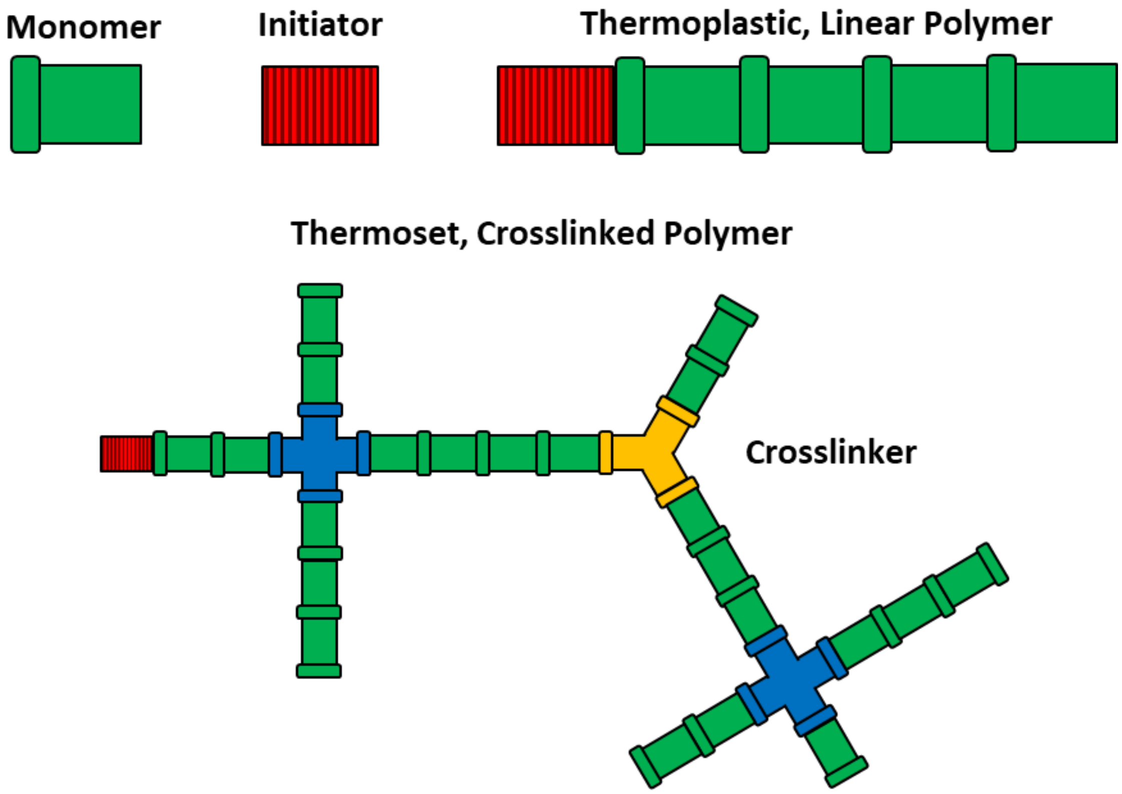

Figure 2. Pipe metaphor for polymers.

Polymers are everywhere and often at the center of environmental news. Large amounts of plastic and textile waste and limited biodegradability and recyclability have become significant environmental issues. However, these big problems have also yielded big innovations, including biodegradable polymers and recyclable thermosets. Other innovations include high performance and stimuli-responsive materials.

6.4 Defining "3D Printing" and Its Significance

"3D printing" or "additive manufacturing" (AM) creates objects by successive layer-by-layer deposition of material as opposed to "subtractive manufacturing" where material is stripped from a block (think sculpting). The benefits of 3D printing include complex geometries, customization, prototyping, and reduced waste. There are complex geometries, like those containing many voids or interlocking parts (Figure 3), that are inaccessible through filling a traditional two-part mold. 3D printing is often used to make custom objects that only need to be made once or even a few times, like a retainer or prosthetics. That way, there is no need to waste material on making a mold. Furthermore, because no mold needs to be made, it enables rapid prototyping of a design before it is finalized. 3D printing has exploded in innovation and accessibility in the last few decades. Printers are compact and hobbyists can purchase one for under $200.

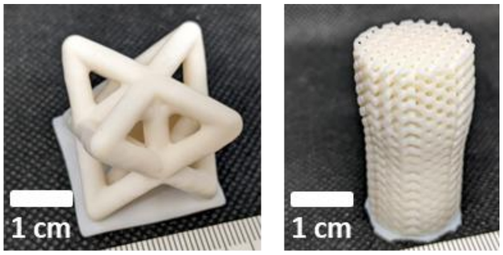

Figure 3. Complex geometries only accessible through 3D printing. Left: octet truss unit cell. Right: gyroid.

The broad types of polymer 3D printing are extrusion, resin, powder bed fusion, material jetting, and binder jetting (Figure 4). Extrusion printing involves melting a thermoplastic to be dispensed thinly to draw each layer of the part. Resin printing uses light to polymerize, or "cure," liquid resin into a solid object. Powder bed fusion uses a laser to sinter, or heat, powder into a single object. Material jetting deposits droplets of liquid polymer, then applies light to instantly cure the liquid to solid. Binder jetting applies a liquid bonding agent to layers of powder to bind the powder together to form connected layers.

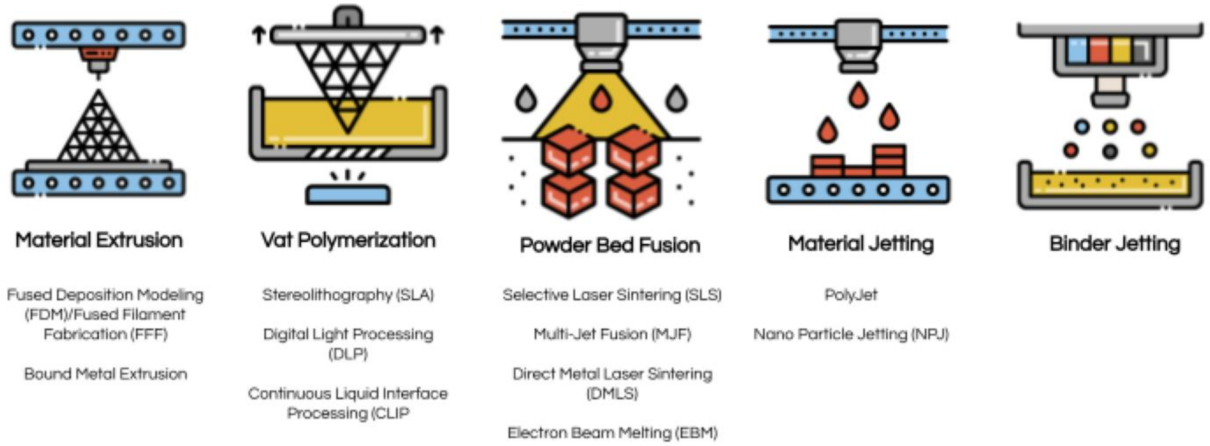

Figure 4. Types of polymer 3D printing.[1]

I used resin 3D printing for my projects, specifically vat photopolymerization (VP, Figure 5). The resin contains monomer(s) and/or oligomer(s), which are short chains of monomers, crosslinker, and photoinitator, which is an initiator that is activated by light. The resin is poured into a vat with a transparent bottom and a build plate, which is a flat plane, is lowered into the vat, almost touching the bottom. The distance between the build plate and the bottom is the layer thickness for the print. A light source, like a projector or a liquid crystal display (LCD) screen, projects the 2D sliced image of each layer of the 3D object sequentially onto the transparent bottom of the vat. The build plate moves up and each 2D projection cures a layer of the resin from liquid to solid. Thus, the object is printed upside down from bottom to top.

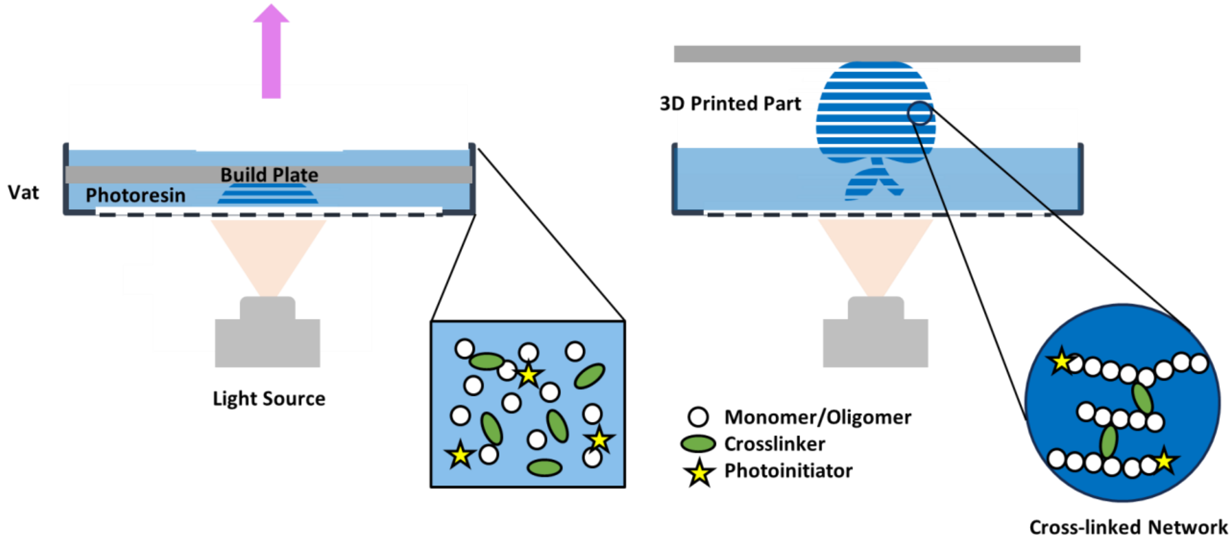

Figure 5. Vat photopolymerization (VP) diagram.

6.5 How I Chose My Research

Prior to UW, my research experience was in bioanalytical chemistry, where I used various instruments to study chemistry related to living things. I studied origins of life in extreme, Mars-like environments and quantified fatty acids and glucose in the human body. I analyzed things that already existed and I saw my graduate career as an opportunity to create something new. As a materials chemistry student, I began by taking one hard materials course and one soft materials course, which was polymers. I was fascinated by the simple, repetitive chemistry and the ability to create a wide variety of materials that we use every day. While getting to know the Boydston group, I had the opportunity to try 3D printing and I was very excited by the ability to so quickly make something that could be used.

6.6 My Research

My research centered on bringing new abilities to 3D printed materials. In one branch of projects, I aimed to make self-strengthening materials and in the other, I aimed to print circuit boards. Being able to print these materials unlocks complex geometries that are inaccessible through molding and rapid prototyping.

6.6.1 Self-strengthening Materials

Many materials undergo degradation, but few adapt to their degrading conditions. Bone is an example of a material that can adapt: it can add new material to areas that experience stress. This has motivated the development of self-strengthening materials. By incorporating self-strengthening abilities into 3D printed parts, these geometries can be designed to better withstand stress and be made with less material. For example, if this chemistry were incorporated into the structure of a bridge, after certain parts of the bridge were damaged by stress, those parts could restore their strength.

The overall process involved 3D printing a small cylinder (forming the primary crosslinked network), swelling it with a pre-strengthening solution, then applying strain to activate the strengthening (forming the secondary crosslinked network), resulting in a stronger cylinder. The 3D printed cylinder was a relatively weak material (specifically, it was easily compressed). How did the material "know" when to strengthen and how did it strengthen? In addition to the necessary components listed in section 6.4, this 3D printing resin contained piezoelectric particles, which were the key component that enabled self-strengthening by factors of 1.2-2.2. Piezoelectric particles convert mechanical energy to electrical energy. This electrical energy can initiate polymerization. By swelling the cylinder with liquid monomers, the object held the building blocks for a second polymer network. When mechanical energy, or stain, was applied to the object, electrical energy was generated, initiating the formation of the secondary polymer network. This final object now had the combined strength of the primary and secondary polymer networks. You can think of the primary network like the original bone and the secondary network like the added material to bone. Now the original structure is both repaired and even stronger than before.

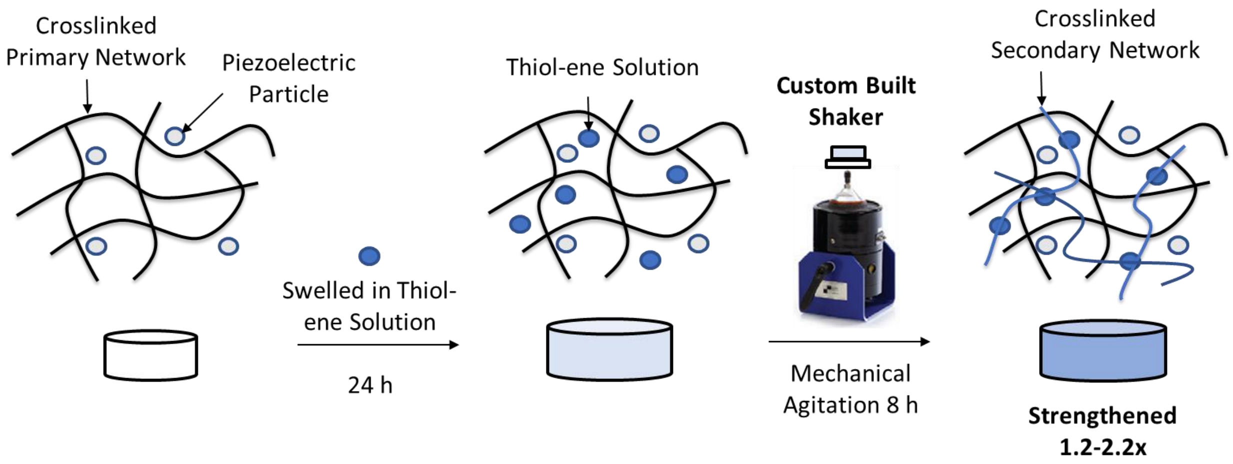

Figure 6. Piezoelectric self-strengthening process overview.

The main challenges with 3D printing the piezoelectric particles were suspending them in the resin and printing at high resolution, or detail. It is important to suspend the piezoelectric particles in the resin so that they are evenly distributed throughout the printed object. If the particles sink to the transparent bottom of the vat over time before the print is over, layers that are printed later will contain more piezoelectric particles. If there are too many particles at the bottom of the vat, they will block the light projection from curing enough polymer to adhere to the rest of the object, which results in an incomplete print, or a “cut off” object. To suspend the piezoelectric particles, I included an extremely viscous oligomer that had a consistency like honey in the resin formulation. To test the amount of time the particles were suspended, I mixed different amounts of viscous oligomer and non-viscous monomer that flowed more like water, then visually monitored the piezoelectric particle settling over time (Figure 7a). The non-viscous monomer was included because it had a quick cure time and ability to print with high resolution. The other challenge the particles present is that because they are opaque and white, they reflect the projected light. This causes curing beyond the projected image, which results in blurry details. You can think of this like coloring outside the lines. To adjust for this, I shortened the cure time, or how long the light is projected for each layer. However, a cure time that is too short means that the layers may not stick to each other and result in an incomplete print (Figure 7b). I found the precise cure time to get excellent resolution parts (Figure 7c).

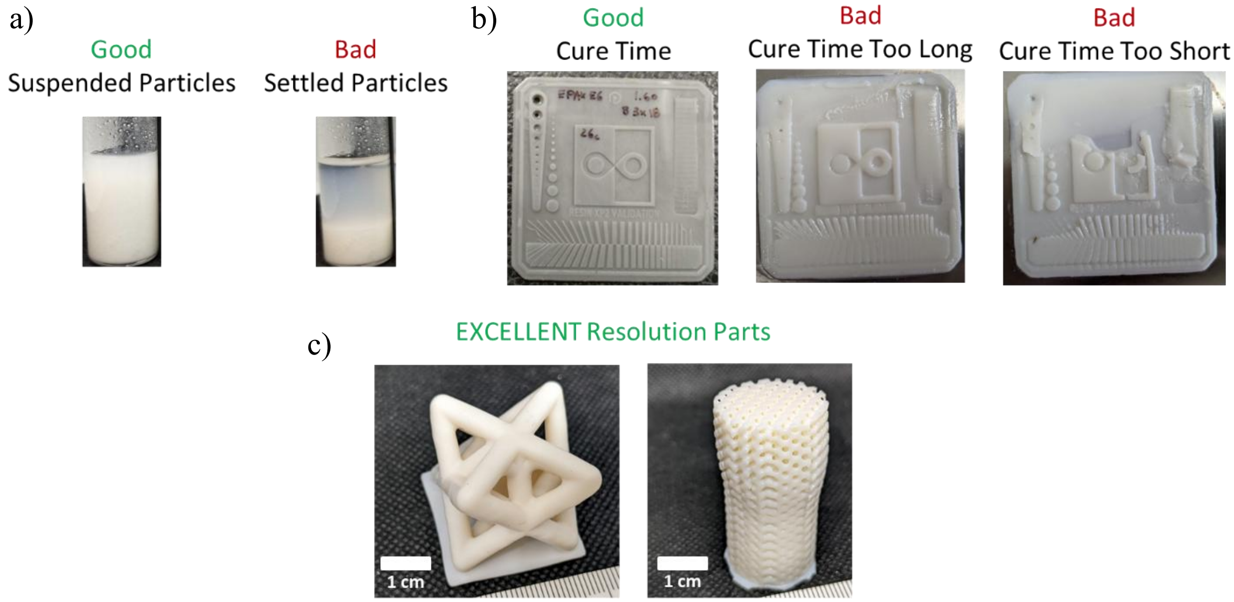

Figure 7. 3D printing parameter optimization: a) settling time of the piezoelectric particles, b) cure time, c) printed parts after optimization.

The main challenges with the strengthening part of the process were choosing a solvent and an agitation method. The solvent needed to swell the 3D printed cylinder like a sponge to get the pre-strengthening components penetrated completely into the part without overswelling it such that it experienced damage. Several solvents were tested to hit that sweet spot. You can think of it like filling a balloon with as much water as you can before it breaks. The agitation method needed to exert continuous strain on the cylinder. More stress experienced by the part meant more strengthening. I tested several different setups and shaking patterns to get the most stress on the cylinder.

The cylinder's strength was quantified using compressive dynamic mechanical analysis (DMA), which squeezes the cylinder and measures the resulting deformation. Imagine squeezing a stress ball versus a tennis ball. The tennis ball requires much more force to squish than the stress ball. On a smaller scale, the cylinder before strengthening was like the stress ball and the cylinder after strengthening was like the tennis ball. In the end, I was able to incorporate a self-strengthening ability by about a factor of two into 3D printed parts. This enabled them to strengthen to be even stronger than they were originally after experiencing damage by shaking. This type of technology can be incorporated into architectures like bridges, vehicles, and anything that experiences stress by vibration.

6.6.2 Printed Circuit Boards

Resin 3D printing objects that are composed of more than one material usually requires multiple vats, each with different resins, or a fluidic exchange system (Figure 8). Because this method prints objects layer by layer, these methods can only change materials in one direction, moving bottom to top. So, you could print a striped apple, but not a polka dotted apple. Additionally, physically swapping out the resins means these methods are slow and require a lot of cleaning. Metal and polymer 3D printing share some techniques, but these materials are printed separately. In this project, I aimed to 3D print circuit boards made of conductive metal and insulating polymer from a single vat of resin, enabling efficient, complex patterning of two different materials.

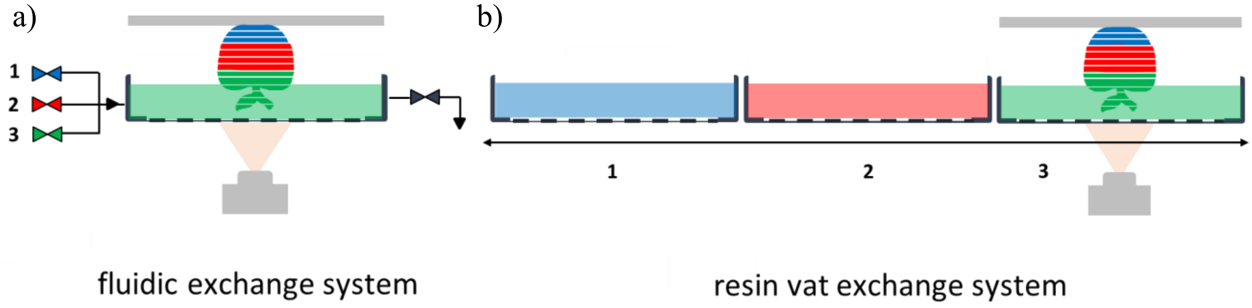

Figure 8. Existing multimaterial 3D printing methods: a) fluidic exchange system and b) resin vat exchange system.

Our team of researchers from the University of Wisconsin-Madison, University of California, Berkely, Lawrence Livermore National Laboratory, U.S. Army Research Laboratory, and Analog Photonics developed a single resin from which metal and polymer can be printed simultaneously using two different colors of light to drive separate chemical reactions that form each type of material (Figure 7a). There are two keys to this process: (1) All of the building blocks to form the metal and polymer are in the resin and do not react with each other until they are irradiated with light. (2) The colors of light used to activate each chemistry and the two chemistries themselves do not overlap. The building blocks for the metal are a metal salt and a photocatalyst. The photocatalyst accelerates the transformation of the metal salt into metal when the light is shining on it. The building blocks of the polymer are monomer, crosslinker, and photoinitiator. The photocatalyst and the photoinitator need to be activated with two separate colors of light. I tested a variety of photocatalysts and photoinitiators to ensure this separation. I also evaluated the resin's stability, ensuring resin components did not react with each other unless they were exposed to those key colors of light.

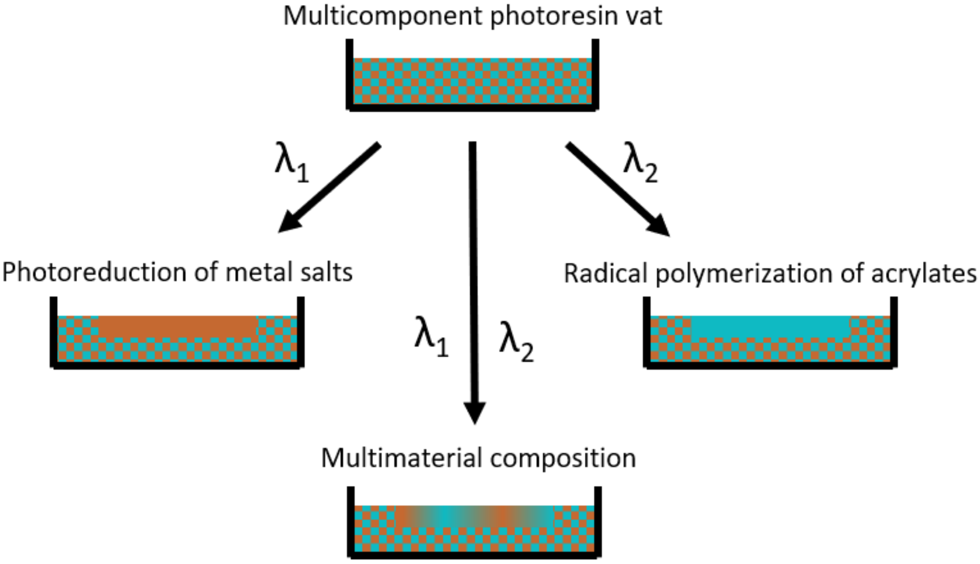

Figure 9. Multimaterial single vat 3D printing of conductive metal and insulating polymer.

Comparing different 3D printing methods, there is typically an inverse relationship between resolution and speed. Although volumetric 3D printing has the fastest speed and lowest resolution, we built a new printer to maintain the speed and increase the resolution to get an unprecedented combination of high speed and resolution. This will enable efficient commercial production of tiny circuit boards.

6.7 Takeaways

I hope that this chapter has taught you some science, made you want to pay greater attention to scientific breakthroughs, and opened your mind to connections between science and other fields. Personally, I have expanded both the breadth and depth of my knowledge during my PhD. I have become a better scientist, communicator, mentor, and mentee. Most of all, I have the depth of my persistence. Next, I will be a Senior Product Developer studying adhesives at 3M in St. Paul, MN.

6.8 Acknowledgement

Thank you to the Wisconsin Initiative for Science Literacy for encouraging this effort to make thesis work more accessible and specifically Elizabeth Reynolds for her feedback and Cayce Osborne and Bassam Shakhashiri for their support.

6.9 References

(1) Additive manufacturing – General principles – Fundamentals and vocabulary. International Organization for Standardization, **2021**. ASTM/ISO 52900:2021.