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Synthesis of Metal Powders for Advanced Additive Manufacturing

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Motivation

Metal additive manufacturing methods often involve localized sintering of powders. Lack of particle size control and the need to apply very high temperatures and temperature gradients to fuse material can lead to degraded spatial resolution and mechanical properties. We use chemical methods to additively manufacture the powders themselves, affording greater control of particle size. We can also make nanoporous particles or coatings with substantially higher surface area and surface energy when compared to their nonporous counterparts. The high surface energy is expected to facilitate sintering at reduced temperatures and improve product quality. It is desirable that our synthesis methods be scalable in order to produce usable quantities of powders.

Electroless Plating

"Electroless" plating is a widely used method where the substrate catalyzes reaction between a metal salt and reducing agent to deposit the metal on the substrate. The reducing agent often contaminates the product, and the growth rate and extent can be difficult to control. Tailored porosity is not easily achieved.

Our approach: Iterative Synthesis

We exploit the fact that palladium hydride can be a reducing agent. We form the hydride by exposure to hydrogen gas:

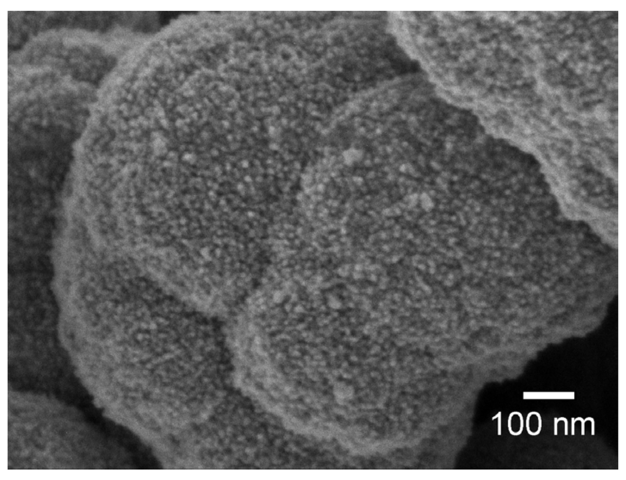
$$\mathrm{Pd} + 0.3\mathrm{H}_2 \rightarrow \mathrm{PdH}_{0.6}$$

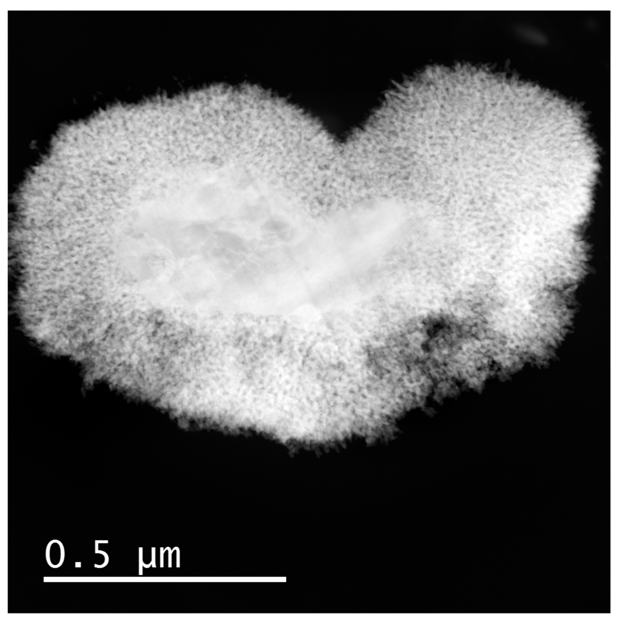
Then we react the hydride with tetrachloropalladate ion in a surfactant solution:

$$3.3\mathrm{PdH}_{0.6} + \mathrm{Na}_2\mathrm{PdCl}_4 \rightarrow 4.3\mathrm{Pd} + 2\mathrm{NaCl} + 2\mathrm{HCl}$$

The process can be iterated (and automated) to grow layers of precise thickness. The process is exponential with each step, allowing the process to transcend length scales.

Results: Nanoporous Coatings



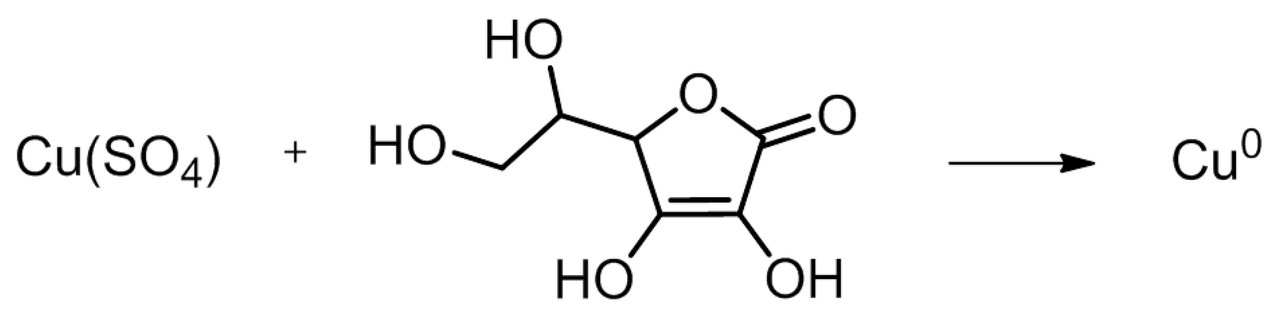


Nanoporous Pd can be grown on nonporous Pd particles to increase surface area for chemical reactions at the surface.

Galvanic Displacement

The process of galvanic displacement uses a less noble metal as a sacrificial template for the synthesis of a more noble metal. This is often carried out by exposing the less noble metal to an aqueous salt solution, subsequently reducing it to yield the desired metal. The "templating" effect allows a reduced metal to retain many of the geometric characteristics as that of the sacrificial metal, thus providing exceptional control over shape and size of the final product.

We can synthesize Pd particles with a high degree of uniformity and porosity by using nonporous Cu particles as a sacrificial templet. Nonporous Cu particles can be initially synthesized by reduction of $Cu(SO_4)$ with sodium ascorbate.

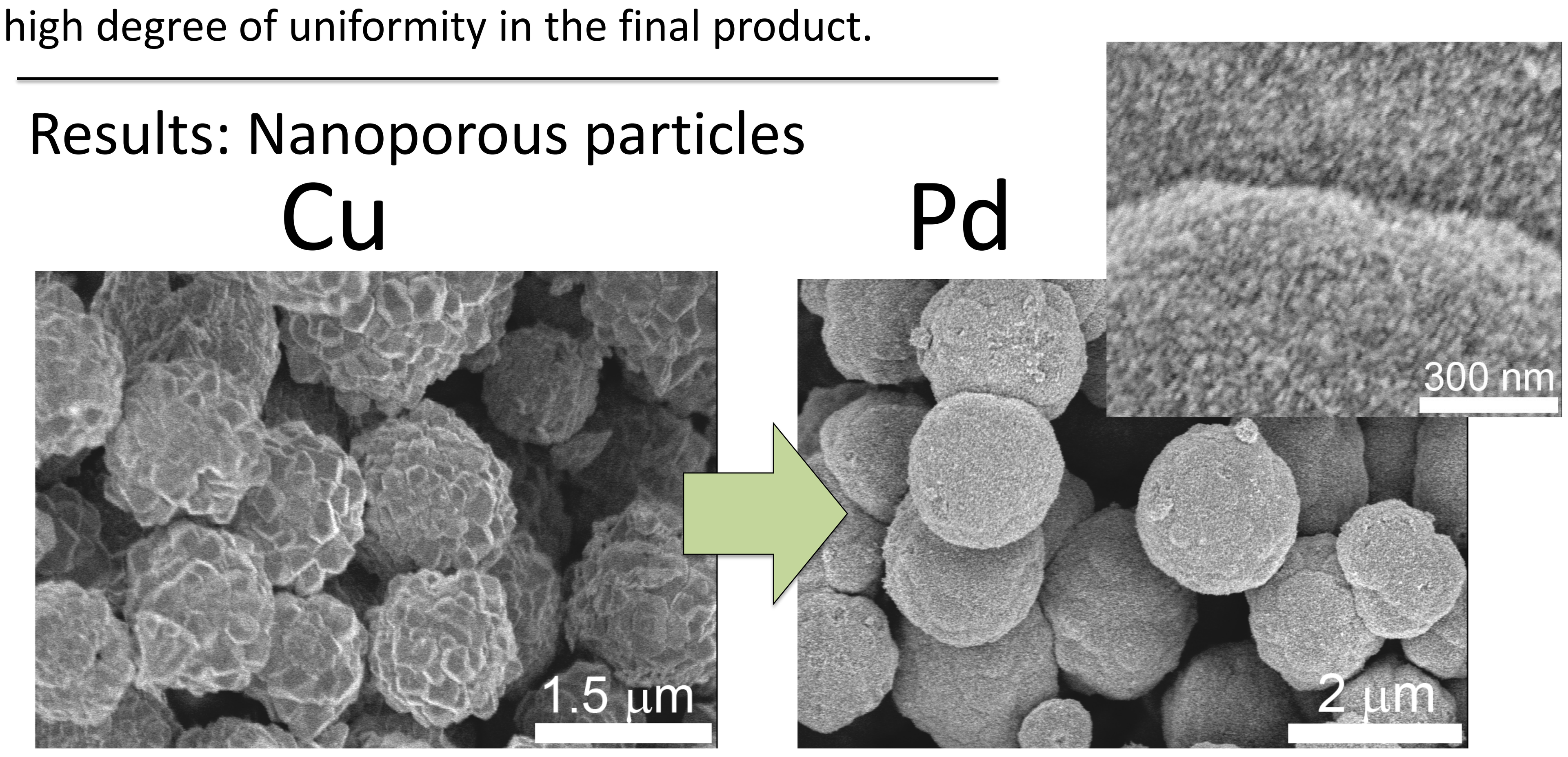


We use the synthesized Cu particles as a solid-phase reducing agent to react a solution containing a tetrachloropalladate ion and surfactant:

$$Cu^0 + Pd^{2+} \rightarrow Pd^0 + Cu^{2+}$$

The reaction occurs at room temperature over a 24 hour period and can easily be scaled to produce gram scale quantities of powder while still maintaining a high degree of uniformity in the final product.

Results: Nanoporous particles



The reaction is geometrically self-limiting, preventing uncontrolled growth of the reduced metal particles.

Conclusions and Outlook

We have developed two methods that allow for precise tailoring of the particle size and surface area of metal powders. We have used palladium due to its convenient chemical reactivity in aqueous solution under ambient laboratory conditions, but similar chemical reactions are known for lower-cost first-row transition metals. We believe that these powders will lead to improvements in the spatial resolution and mechanical properties of 3D printed structures, and aspire to test this hypothesis in the near future.

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