

Precursor development of iron nanopowders for nanoink Aerosol Jet 3D printing

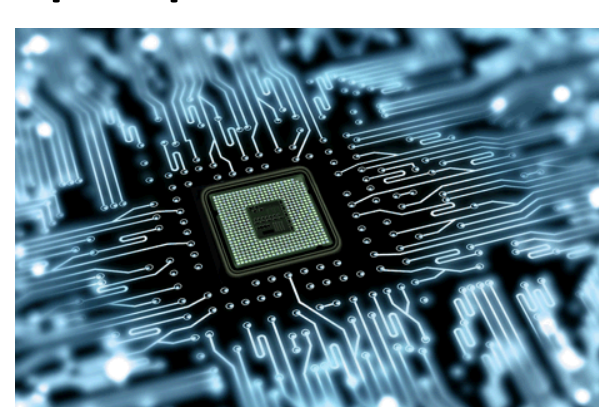
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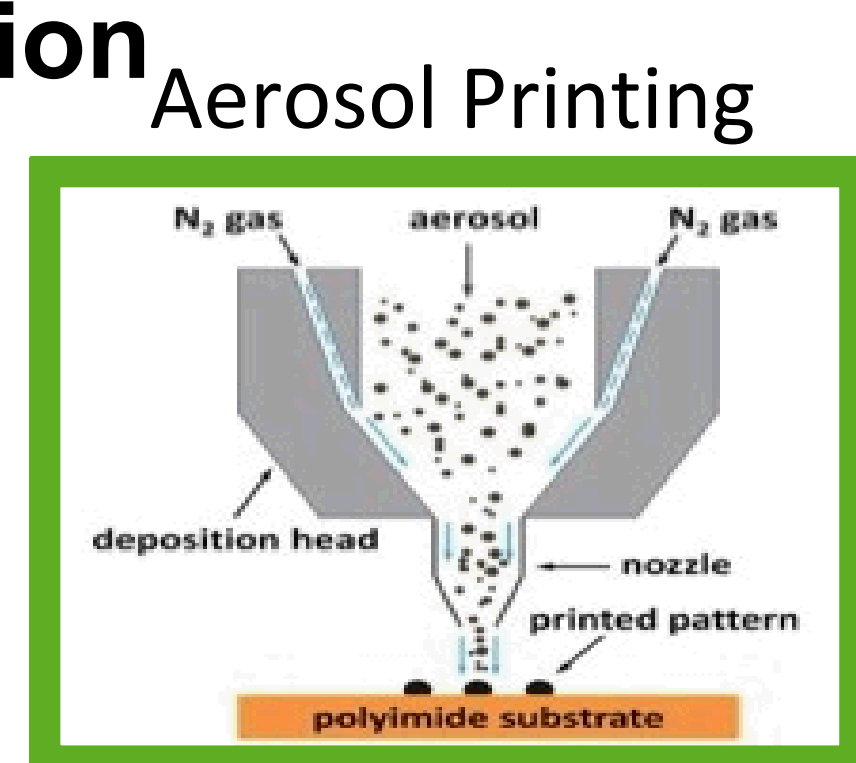


Introduction

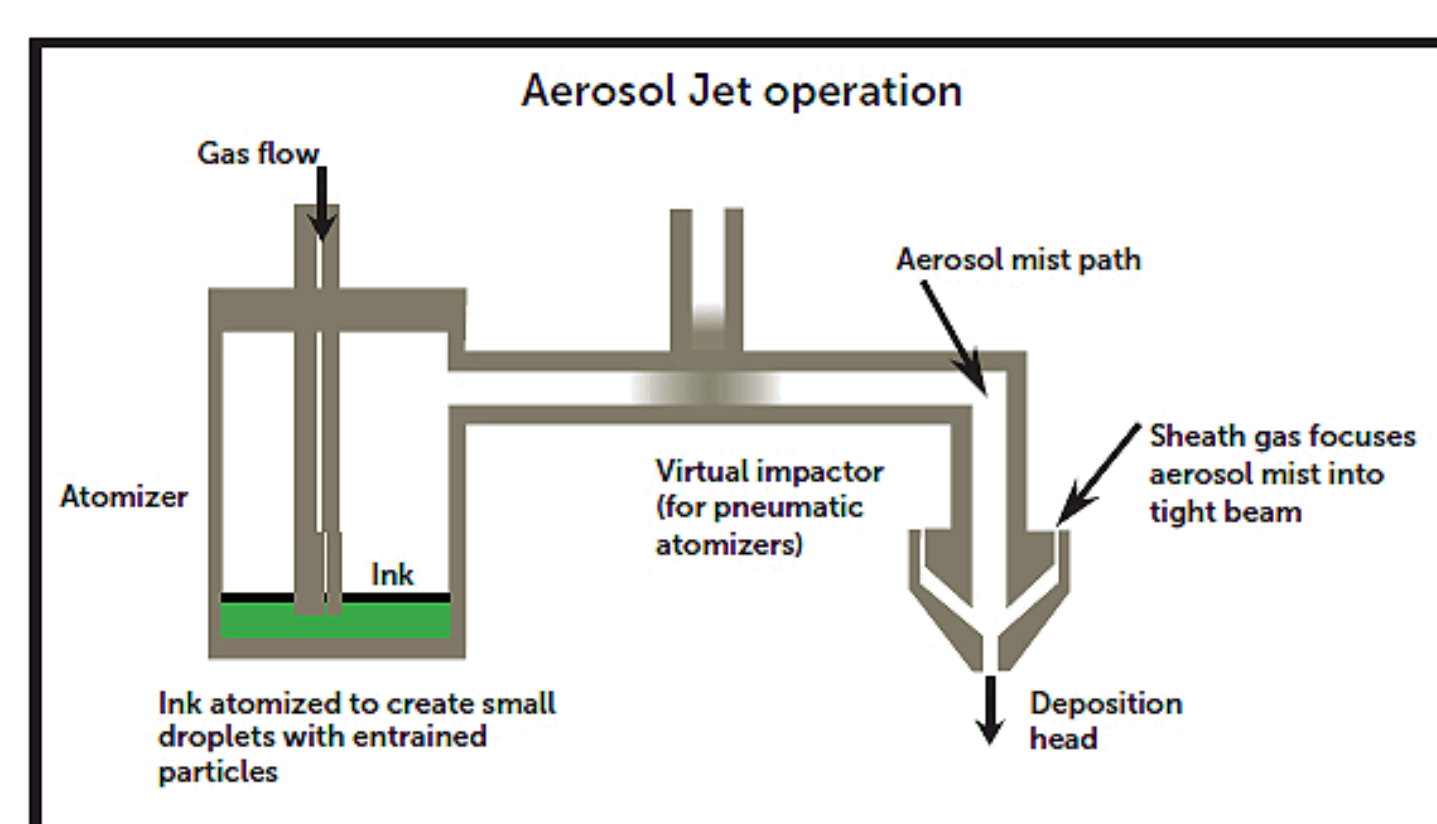
Direct Write (DW) manufacturing methods can precisely print microcircuits, computer chips, and other electronics. Currently, electronics printed by DW methods have focused on silver and gold components due to their high conductivity. However, the high cost of these materials has led to the investigation of other more economical metals, such as copper. For Cu, we have successfully generated Cu nanoinks (N-inks) that produce high quality components. Following this success, we have expanded our research to other first row transition nanometals due to their diverse properties.



In particular, we have synthesized nanoiron metal particles, converted them to Fe N-inks, printed traces by DW methods, and tested the electronic and magnetic properties. To generate the Fe nanometals, a variety of tailored precursors such as metal alkoxides, amides, alkyls, as well as commercially available compounds were evaluated as useful precursors to high quality (size, shape, crystallinity) Fe nanoparticles for production of tailored Fe N-inks.



Application



Process

- Aerosolize the ink in atomizer
- Remove excess gas to make mist more dense
- The dense mist reaches the deposition head and an inert gas flow focuses the mist into a tight beam.

Advantages

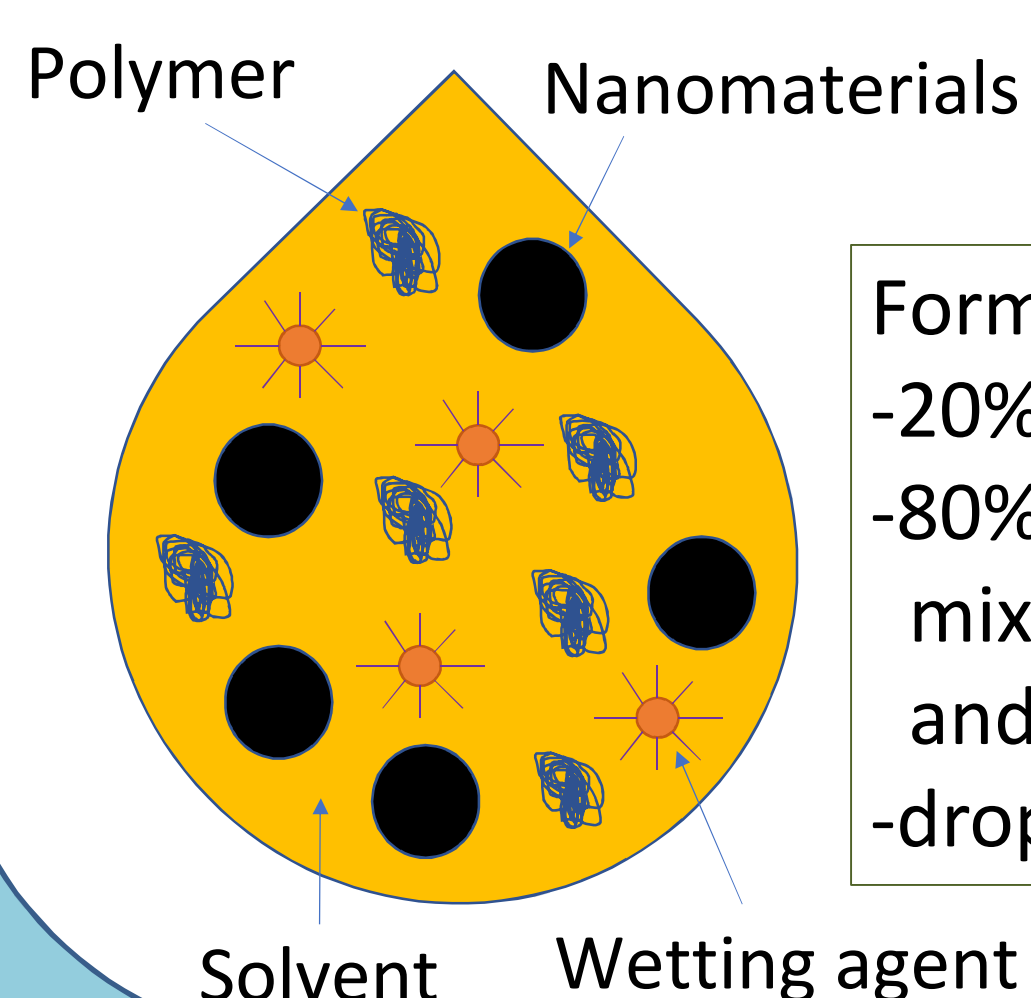
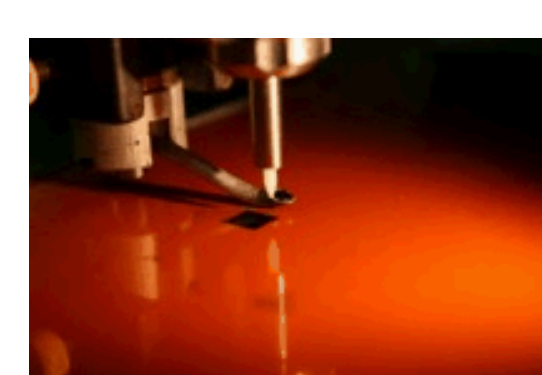
- 1-5 micron droplets
- Continuous stream of high dense ink
- Nozzle does not contact the surface, conformal features printed over a variety of substrates and curved surfaces can be realized.

Ink Formulation

Critical component of Aerosol Jet Printing is the Ink. Starting point is the generation of stable, satellite-free droplets to ensure high-quality printing therefore high quality nanoparticles are critical.

Ink systems are formed from multiple components:

Nanoparticles Solvent Wetting Agents Dispersants Polymers



Formula for making inks:

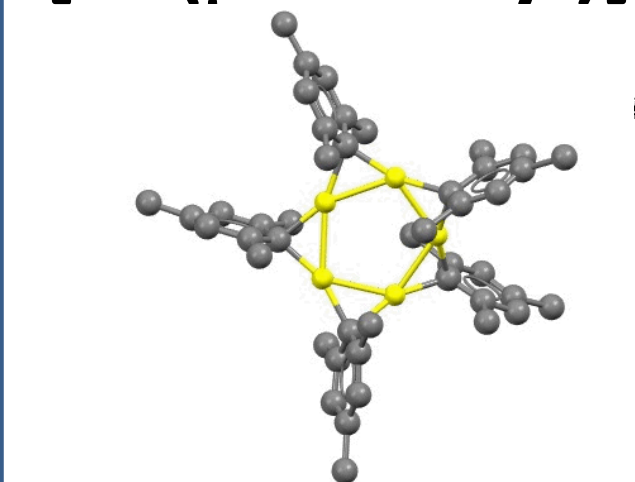
- 20% NP
- 80% solvent mixture (80:20 xylene and white spirits)
- drop of Solsperse 9000

The ink was printed on Kapton. Other substrates usually did not provide a reliable surface for printing due to lack of quality in the print.

Previous work with copper in the lab

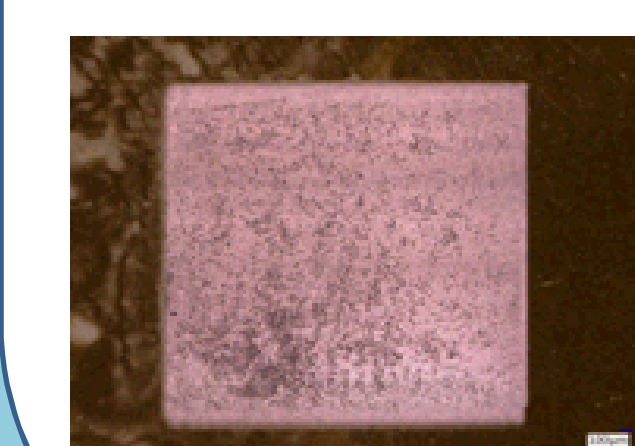
High quality copper nanoparticle (NP) synthesis was idealized for Aerosol Jet Printing at low temperature using copper mesityl with SPPT route.

[Cu(μ -Mesityl)]

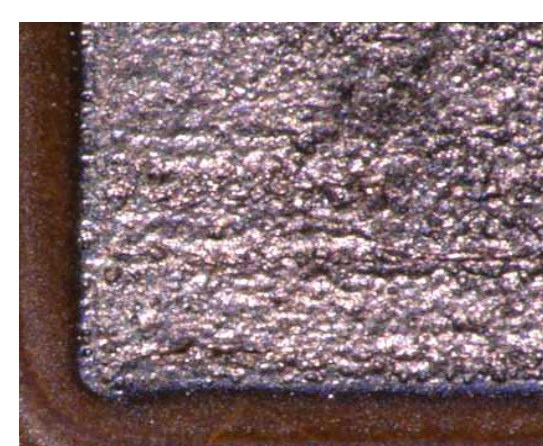


HDA only

NP size ~5-10 nm



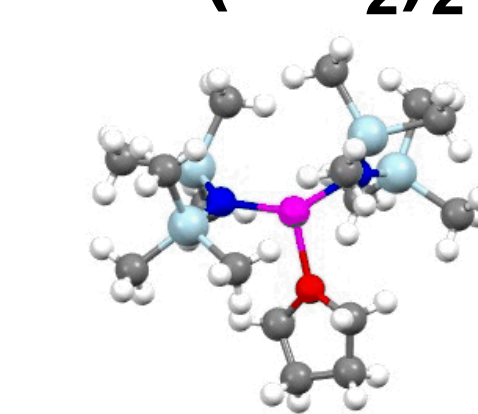
The large scale production of high quality, monodispersed Cu NP supported the formulation of high quality inks.



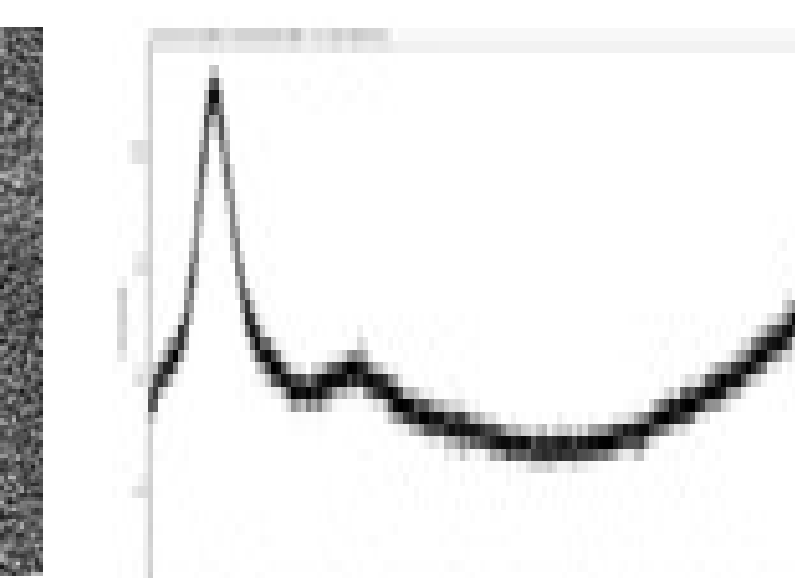
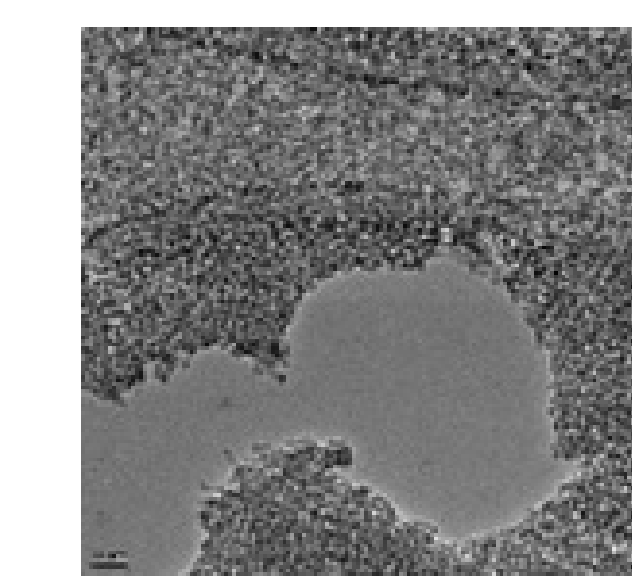
Previous work with cobalt in the lab

High quality cobalt nanoparticle synthesis was idealized for Aerosol Jet Printing using cobalt amide and 8N with SOLVO route.

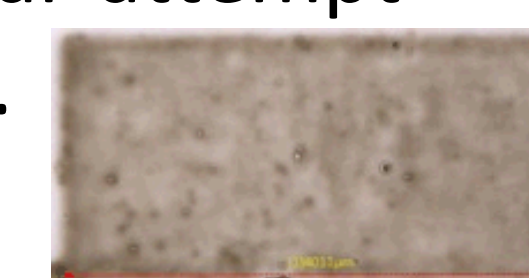
Co(NR₂)₂



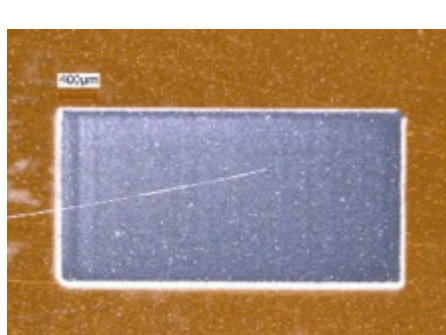
8N



The magnetic Co NPs causes agglomeration and inconsistency upon ink formulation. Initial attempt led to a bad print.



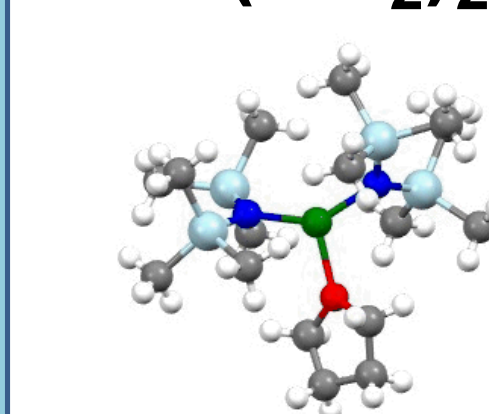
NP size ~10nm
Alternative surfactants were investigated such as OA in attempt to get the nanoparticles fully dispersed which led to a successful print.



Previous work with nickel in the lab

Nickel nanoparticles were synthesized for Aerosol Jet Printing using nickel amide and the use of HDA and 8N with SOLVO route.

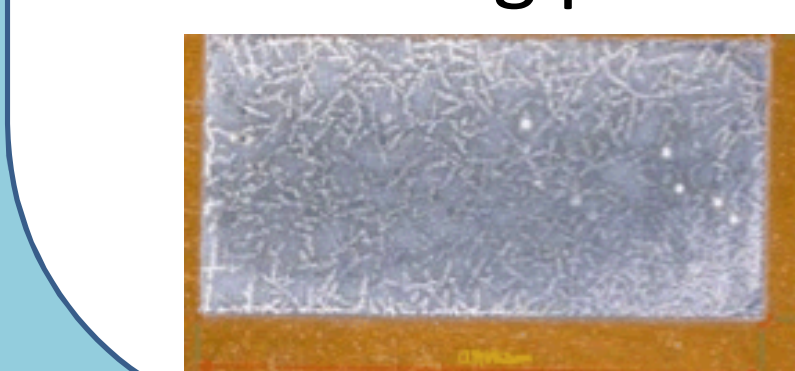
Ni(NR₂)₂



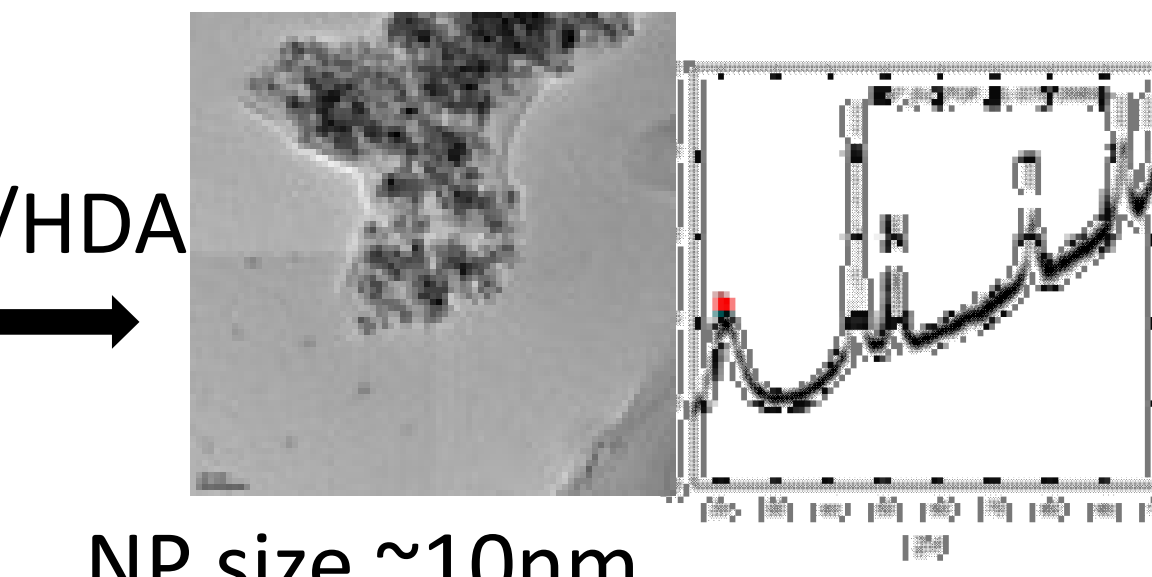
8N/HDA

NP size ~10nm

Pad shows that there is cracking present.



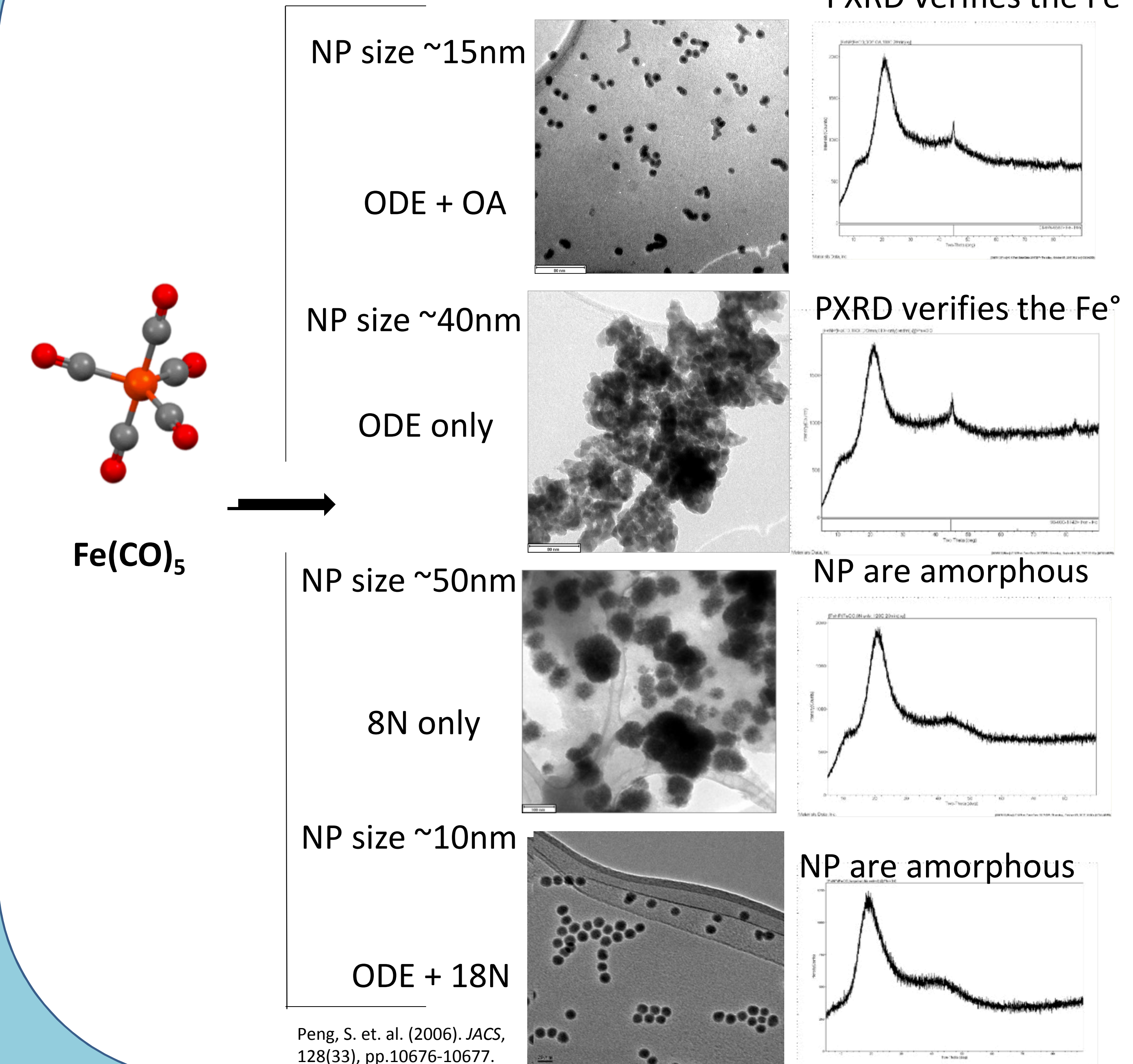
Solvent and heat treatments are still being idealized.



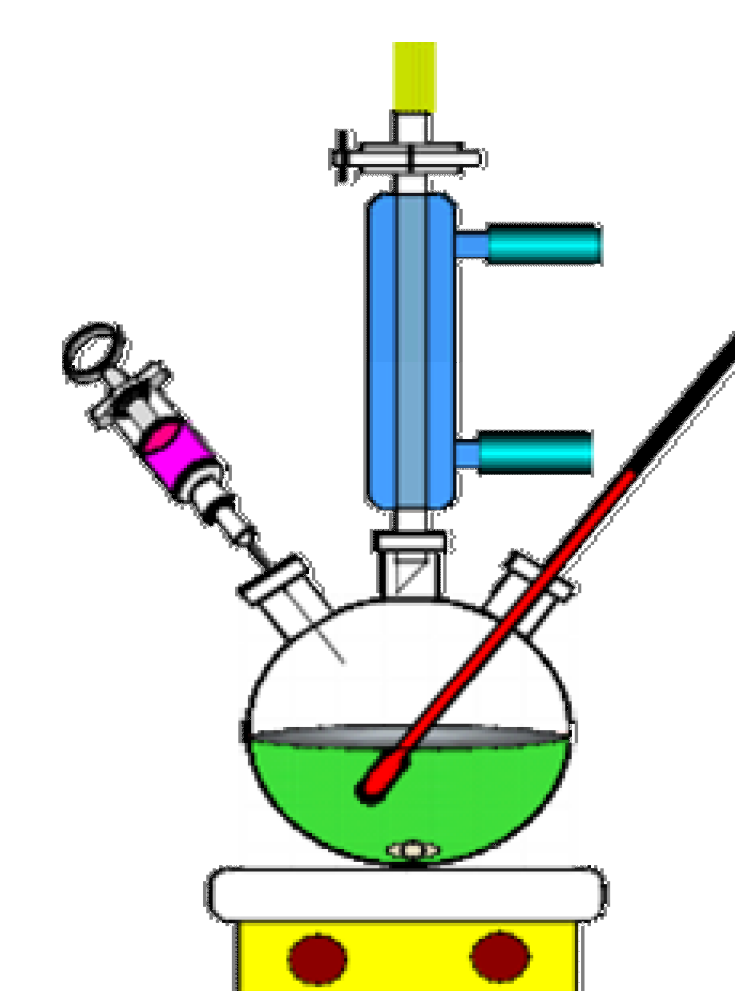
Various surfactants and solvents were investigated with Fe(CO)₅ as a precursor to idealize the synthesis route of iron nanoparticles

Investigating iron nanoparticles and idealizing their synthesis for Aerosol Jet Printing

Additional iron precursors are being investigated to see how precursor effects the morphology of nanoparticles and ultimately the printing.

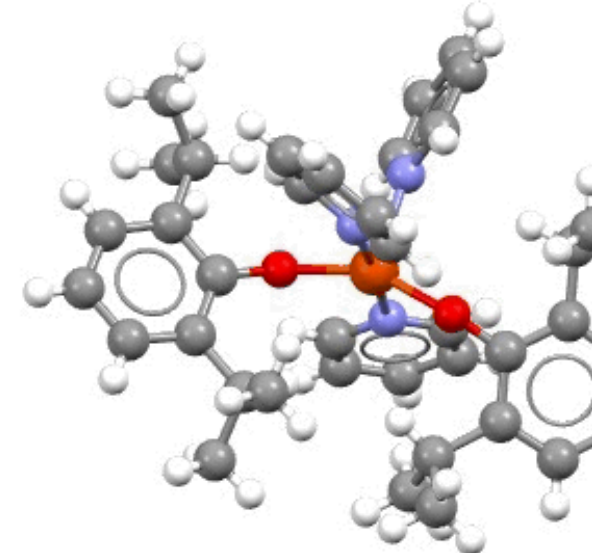


All Fe nanoparticles were made SPPT route. Each reaction was ran at 180°C (120°C for 8N) for 20 minutes. Resulting nanometal product was collected from magnet in reaction and isolated through hexane washes.

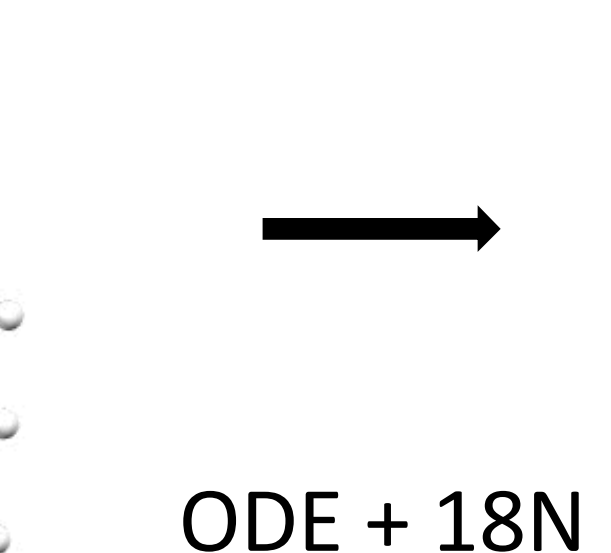


SPPT Set Up

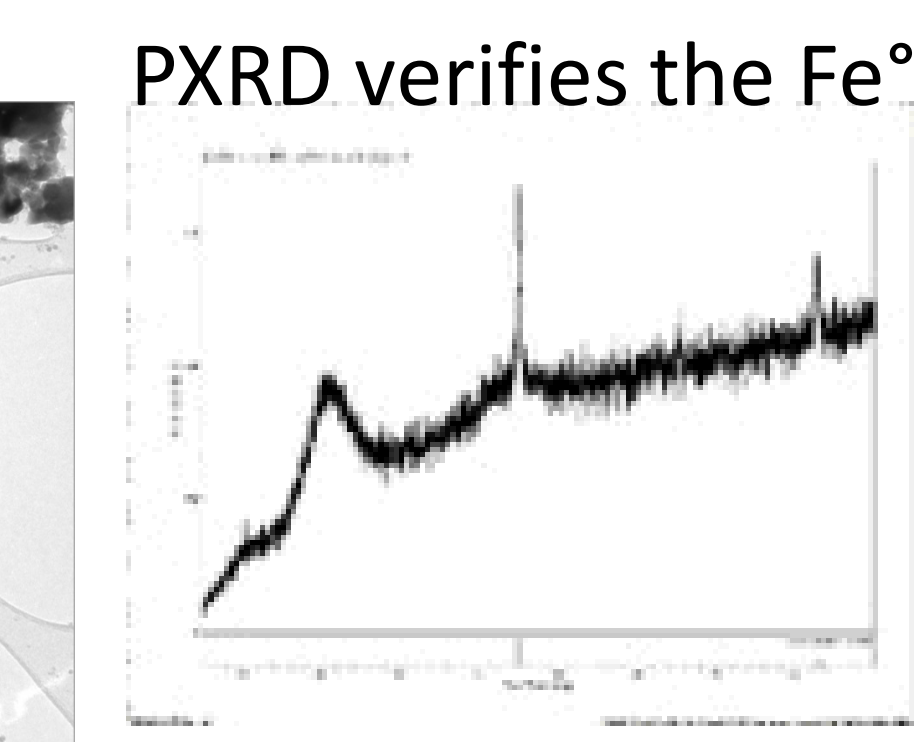
Fe(DIP)₂(py)₃



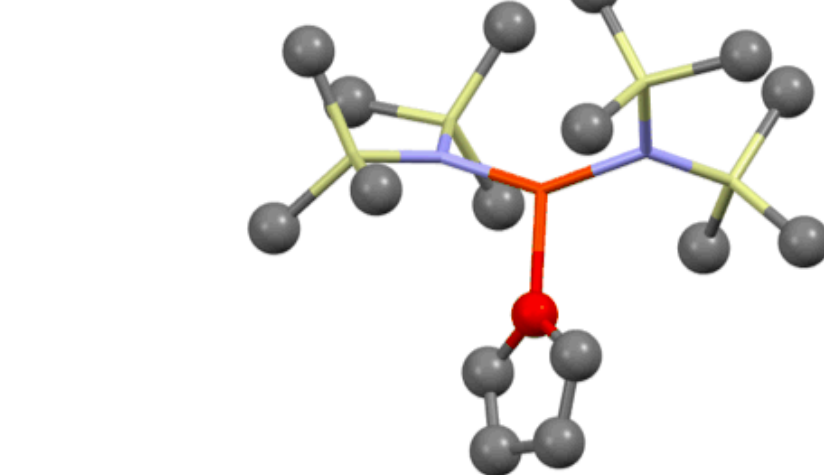
NP size ~50nm



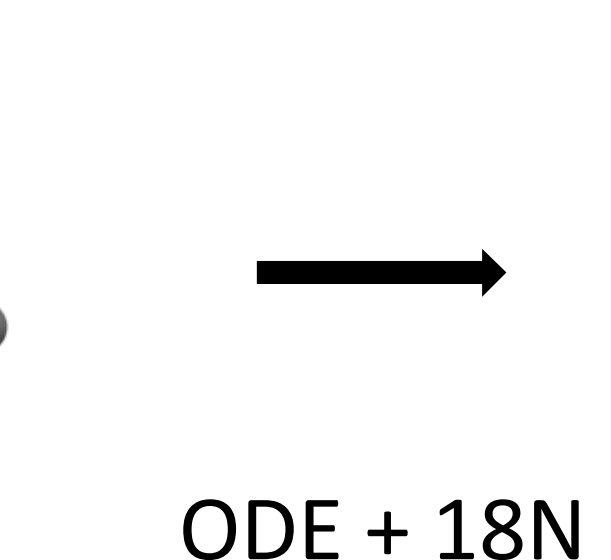
ODE + 18N



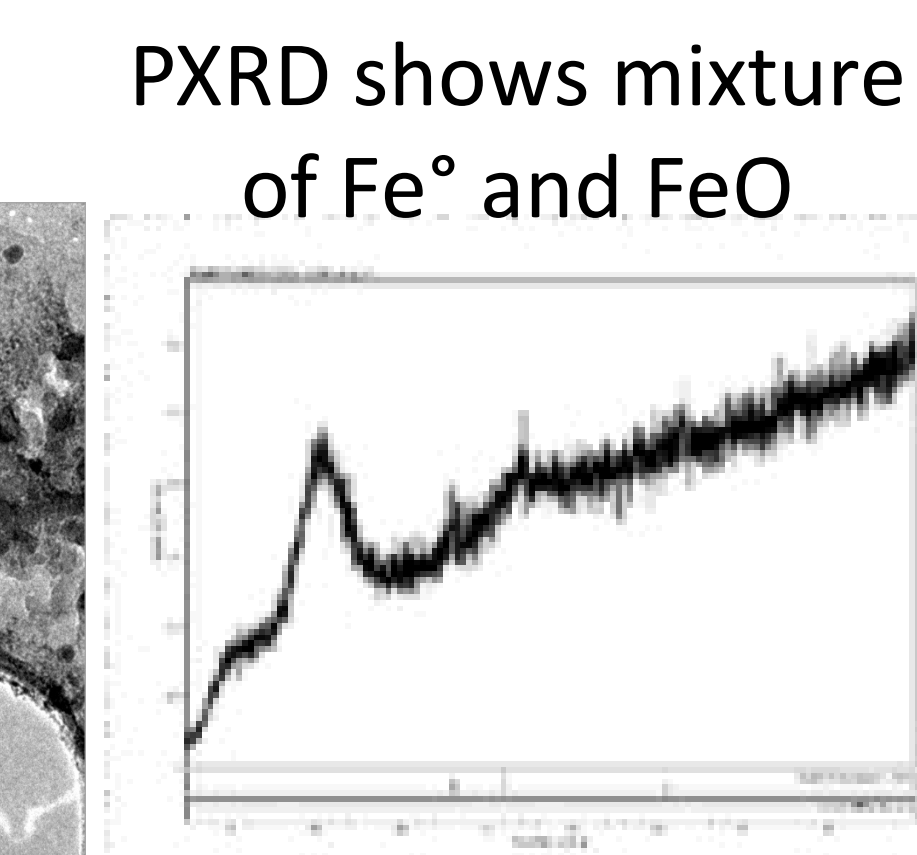
Fe(N(SiMe₃)₂)₂•THF



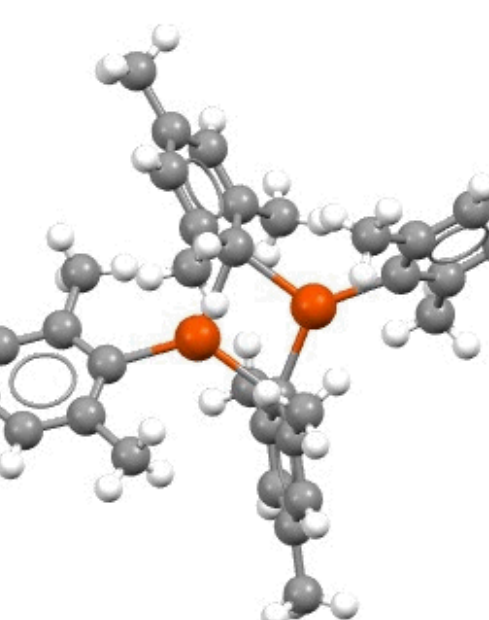
NP size ~10nm



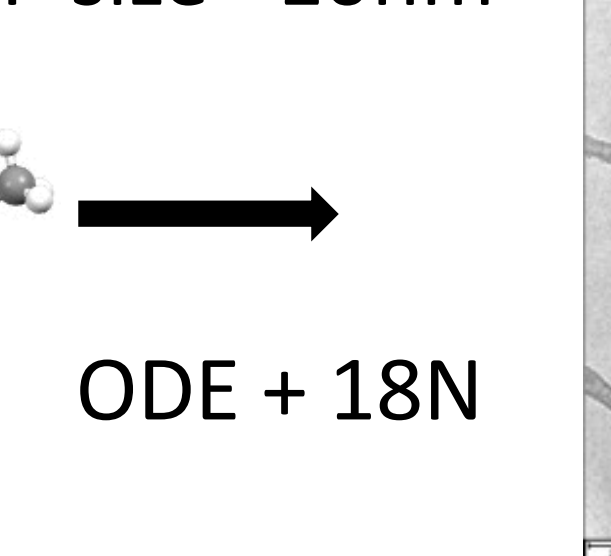
ODE + 18N



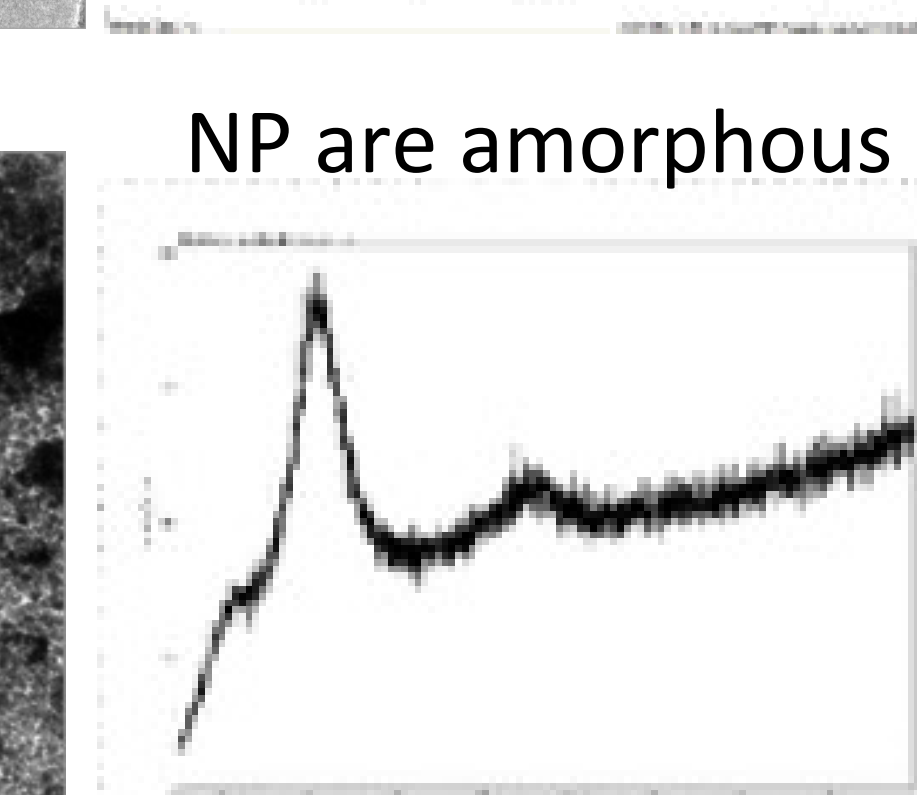
Fe(Mes)₃



NP size ~10nm

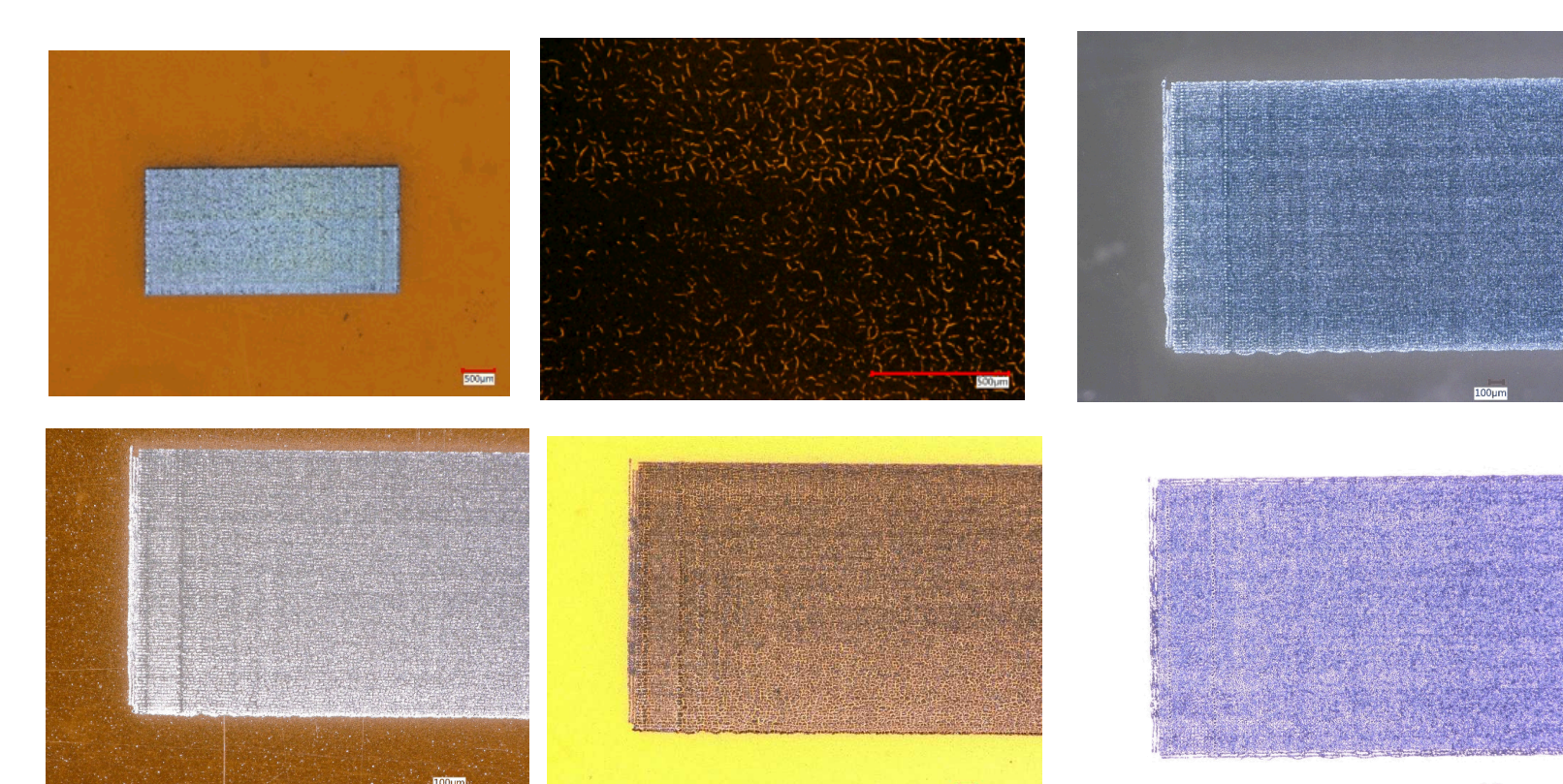


ODE + 18N



Initial Iron Print

Printed pad of Fe using Aerosol Jet™ printing. Post cured images of pads and zooming in cracking is visible with the backlight on.



Kapton

Glass

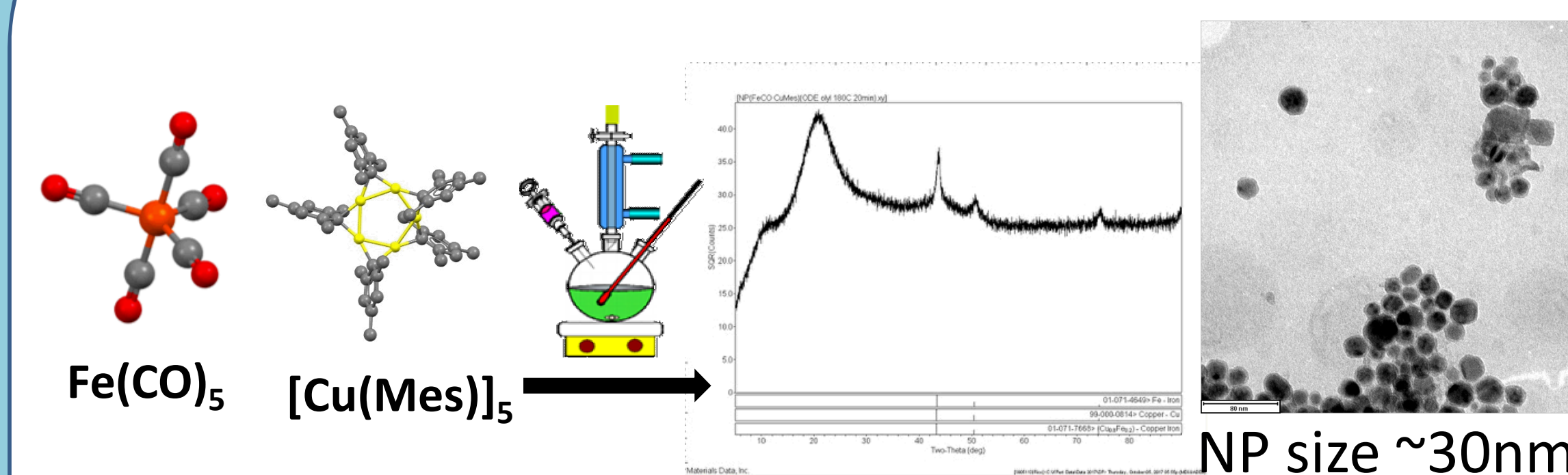
Nanoparticle formation, ink formula, substrate, and curing methods of pads will be investigated to eliminate cracking.

Curing process:

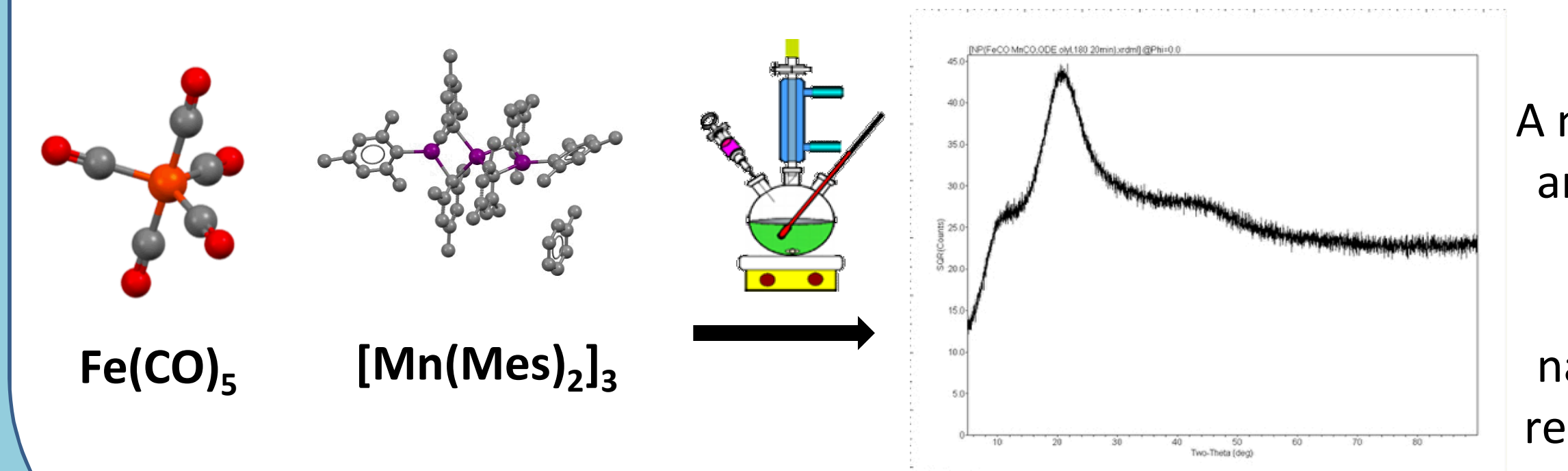
- Tube furnace purged with H₂ (2.9%)/Ar (bal) for 30min.
- Temp set to 375°C and pads placed inside for 30min to cure.



Next Steps: Making Alloys



A mixture of Fe(CO)₅ and CuMes were combined and converted to nanoparticles. Resulting crystalline material reveals it could be Cu, Fe, or CuFe.



A mixture of Fe(CO)₅ and MnMes₂ were combined and converted into nanoparticles. The resulting material is amorphous.

Conclusion

The different phases of NPs provide an opportunity to determine how varying the morphology effects printing.

The magnetic and conductive nature of the different metals offers new capabilities for printed microcircuits and electronic, as well as potential protective shielding for 3D printed electronics.

TEM mapping will be done on the mixed metal material to reveal the composition of the product.

Acknowledgements

Sandia National Laboratories is a multi-mission laboratory managed and operated by National Technology and Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.