

3Y-ZrO₂ Ink for Direct-Ink Writing- User Protocol

Chemicals Needed:

- (1) 3D Printable Yttria-stabilized Zirconium(IV) Oxide Ink (product [918571](#))
- (2) Luperox®, 231, 1,1-Bis(*tert*-butylperoxy)-3,3,5-trimethylcyclohexane (product [479896](#))

Equipment/Consumables Needed:

- (1) Mixer container
- (2) Metal Spatula
- (3) Balance
- (4) Planetary Mixer
- (5) Syringe (with caps)
- (6) Syringe Nozzle
- (7) Syringe plunger
- (8) Direct-Ink Write (DIW) Printer
- (9) Furnace

Step by Step Procedure for Ink Mixing and Loading into Syringe:

- (1) Place mixer container on balance and tare weight.
- (2) Using the metal spatula, scoop out desired amount of the ink ([918571](#)) from container into the mixer container on the balance. *(For reference, for filling a 10 mL syringe, 30 grams of product [918571](#) is sufficient.)*
- (3) Once the desired amount of ink ([918571](#)) is in the mixer container, zero the balance again. Add the Luperox® 231 ([479896](#)), which will be 0.3 wt% of the total mass of ink ([918571](#)) used. *(For example, if 30 grams of ink ([918571](#)) ink is used, 0.09 grams of Luperox® 231, product [479896](#), will be added.)* A plastic pipette works well to add the Luperox® 231 ([479896](#)), dropwise into the ink ([918571](#)).
- (4) After the addition of Luperox® 231 (product [479896](#)), use the metal spatula to mix the ink by hand until the paste is visibly uniform. Viscosity of ink mixture should decrease slightly after the addition of Luperox® 231 ([479896](#)).
- (5) Using a planetary mixer, mix the ink using the following mixing sequence:
 - a. Mix setting @ 2000 rpm for 2 min.
 - b. Defoam setting @ 2200 rpm for 3 min.Repeat this mixing sequence twice. With hand mixing in between.
- (6) Once thoroughly mixed, the ink can be loaded into a capped syringe for DIW Printing. Using the metal spatula, scoop the ink from the container and deposit on the side of the syringe. Carefully tap the ink from the syringe wall to fill the syringe fully.
- (7) After all ink is loaded into the syringe. Place the syringe into the mixer and use the Defoam setting @ 2200 rpm for 10 seconds to eliminate trapped air bubbles that will cause inconsistencies in printing.
- (8) After plunger is placed into the syringe, ink is ready for printing.

Other Notes for DIW:

- Syringe Nozzle sizes ranging from 250 µm – 800 µm can be used.
- Initial pressure for DIW printing ranges from 12-18 psi, depending on nozzle diameter used.

Post Processing of DIW printed parts:

- (1) Curing @ 110°C for 12-16 hours.
- (2) Sintering using the following sequence:
 - Starting at RT, Ramp to 200 °C for 6 hours.
 - Ramp to 300 °C @ 1 °C/min. Hold for 4 hours.
 - Ramp to 400 °C @ 1 °C/min. Hold for 2 hours.
 - Ramp to 800 °C @ 2 °C/min. Hold for 4 hours.
 - Ramp to 1090 °C @ 2 °C/min. Hold for 15 hours.
 - Ramp down to 50 °C @ 2 °C/min.