User Protocol for Ultra-high temperature Ceramic (B₄C) Ink for Direct-Ink Writing

Chemicals Needed:

- (1) 3D Printable Ultra-High Temperature Boron Carbide Ink (product 921912)
- (2) De-Ionized water

Equipment/Consumables Needed:

- (1) Mixer container
- (2) Plastic Spatula
- (3) Balance
- (4) Planetary Mixer
- (5) Syringe (with caps)
- (6) Syringe Nozzle
- (7) Syringe plunger
- (8) Direct-Ink Write (DIW) Printer
- (9) Glass slides
- (10) Oven and Furnace for drying and sintering.

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

- (1) Place mixer container on balance and tare weight.
- (2) Using the spatula, scoop out desired amount of B₄C ink (product <u>921912</u>) from container into the mixer container on the balance. (For reference, for filling a 30-cc syringe, 46 grams of the B₄C ink is sufficient.)
 - i) Note. Due to high solids loading, it is possible that the ink is too viscous to scoop out due to evaporation of solvent. In that case, add 1-2wt.% of de-ionized water to the mass of ink taken in the container and subject it to think mixing. Adding more than 2wt.% of de-ionized water lowers viscosity greatly to an extent that it cannot be printed.
- (3) 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 1 min.

Repeat this mixing sequence twice. With hand mixing in between.

- i) Note. Mix until a smooth paste is obtained.
- (4) Once thoroughly mixed, the ink can be loaded into a capped syringe for DIW Printing. Using the spatula, scoop the ink from the container and deposit on the side of the syringe.
- (5) Carefully tap the ink from the syringe wall to fill the syringe fully.
- (6) 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.
- (7) After plunger is placed into the syringe, ink is ready for printing.

Other Notes for DIW:

- Syringe Nozzle sizes ranging from 400 μm 800 μm can be used.
- Initial pressure for DIW printing ranges from 45-60 psi, depending on nozzle diameter used.
- The ink can be directly printed on a glass/graphite substrate greased with vaseline.

Post Processing of DIW printed parts:

- (1) The printed sample is subjected to air drying at room temperature for 48h, followed by 80°C oven dry for 8h.
- (2) Pre-heating of dried parts at 1050 °C for 3 h at 5 °C/min ramp rate in a tube furnace with flowing gas composed of 4 vol% hydrogen and 96 vol% argon.
- (3) The parts are subsequently sintered in a high temperature graphite furnace at 2260–2295 °C for 1 h under gas flow.
- (4) The sintering temperature can be predicted by the following phase diagram.

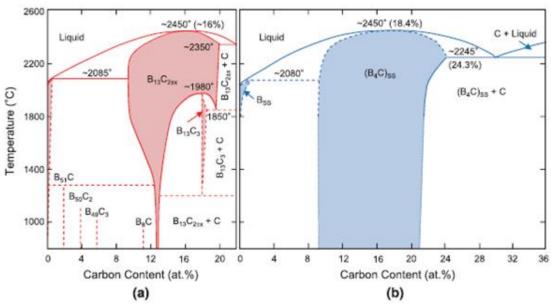


Figure 1: Phase diagram of B₄C (J. Am. Ceram. Soc., 94 [11] 3605–3628 (2011)).