Standard Operating Procedure

Use of Pulsed Electric Current Sintering (PECS) System

Facility: PECS System
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Scope: This SOP details the general procedure for operation of the departments PECS system.

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Introduction:

This Pulsed Electric Current Sintering (PECS) system is a hot pressing system which heats the sample and die by sending a pulsed electric current through the die and sample. This strongly localizes the heating, and provides rapid heating of the sample. The result of such a configuration is the rapid fabrication of pressed samples in minutes as opposed to hours. This helps to minimize grain growth during pressing, and can result in improved mechanical and electrical properties of the samples.

Such systems are often referred to as spark plasma sintering (SPS) systems. Although there is evidence that the current flow through samples can strongly influence the microstructure of certain samples, there is little to no evidence of either sparks, or plasma generation at the grain boundaries [1, 2].

Applications:

PECS is effective for pressing powder materials which can be electrically conductive, or insulating without the use of binders. As the grain size of a material decreases, the strength is known to increase (up to some limit). Thus a smaller grain size is advantageous for increasing strength of samples. Sintering samples at high temperatures for a long time can cause grain growth within samples; however with the relatively fast process of PECS, this grain growth can be minimized. The advantage of short run times also allows for a larger number of sample variations to be investigated in the same amount of time.

PECS has been effectively used for the fabrication of functionally graded materials and samples with near net shapes (through proper design of the dies used).

System and Experiments:

This Pulsed Electric Current Sintering (PECS) system from Thermal Technology, LLC was purchased through the generous support of the Office of Naval Research (ONR). Priority for use of this system is given to ONR sponsored research first. Any publications or presentations resulting from the use of this system should include in the Acknowledgements section:

We wish acknowledge use of the Office of Naval Research DURIP supported Pulsed Electric Current Sintering System at Michigan State University.

Typical Operation:

1) Initial System Setup/Check

- Verify that gas cylinders and regulator valves are open (N_2 – 75psi, Ar – 30 psi)
- Verify that main power disconnect is on
- Turn on chiller
- Verify chiller set point (idle range 65-70F)
- Turn on PECS main breaker
- Turn on the hydraulic system
- Check that gas flow valve is off
- Check that backfill valve is off
- Verify that pump vent valve is closed
- Verify that room exhaust vent is open
- Turn on roughing pump
- Open roughing valve
- Allow the vacuum level to reach vacuum set point (5.0E-2 torr)
- Close the roughing valve
- Backfill chamber
- Open chamber door
- System is now ready to have die and tooling setup in chamber

2) Setup tooling in Chamber

- Use the Ram Enable pushbutton and Ram Jog switch to move the rams to a loading position.
- Inspect the graph foil on the face of each ram. Install or replace as needed.
- If installing new graph foil on rams inspect the ram faces and polish as needed.
- Verify that thermocouple in correctly installed in the lower ram.
- Place die and tooling in correct position in chamber. If using the thermocouple be sure to install in correct position.

- Verify the preload force and punch area are correct for tooling in the Eurotherm controller.
- Slowly use the Ram Enable and Ram Jog switch to apply preload. Be sure that tooling is aligning correctly as force is applied.
- If using the Pyrometer, adjust the pyrometer and die to the correct position.
- Close the chamber door
- Verify that Gas Flow and Backfill valves are closed.
- Open roughing valve
- Allow the vacuum level to reach at min 5.0E-3 torr

3) Turn on Power Supplies:

- Verify that all the front panel circuit breakers are turned on and the green ready light is illuminated on each power supply.
    - Note: the vacuum set point interlock must be meet before ready lights will come on.

4) Turn on PC:

- Start and initiate the Eurotherm iTools software
    - iTools select Scan -> OK
    - Under Views select Program
- Start and initiate the Spec View software
    - Check that Temp control is set to Thermocouple or Pyrometer for experiment
    - Verify the scale and range are appropriate for experiments temperature and time
    - Set log file name to be the experiment number
- Start and initiate the Dynatronix Power Supply Monitor and Recipe Editor software
    - Verify the power supply control pulse settings are 25ms on and 5.0ms off in the Dynatronix software
- Enter the temp and pressure profile for experiment into the iTools software
- Save iTools file as experiment number

5) Starting Run

- Verify that Eurotherm control is configured for correct Thermocouple or Pyrometer control. Select loop button 3 times on 2704 controller
- Verify that punch area is correct. Again select loop X times on the 2704 controller

- Verify that Eurotherm temperature and force loops are set to Auto
- Verify that Eurotherm temperature outputs are at 0%
- Set the chiller set point to proper level for experiment
    - To change set point select Menu twice
    - Use arrows to move set point up or down
    - Press menu to exit
- If experiment is to run under **Argon** environment
    - Close roughing valve and open the back fill valve until chamber is at atmosphere
    - Close back fill
- Turn gas flow on. Set flow meters to mid scale
- Turn Spec View logging on
- Press the Prog button on the Eurotherm to bring up the Program Control window. Press the Prog again to start the run.

6) Monitoring process during the run

- Record experiment start time
- During the run attention should be paid that the system follows the temperature and pressure profiles.

7) Finish run

- Record experiment end time
- Turn Spec view logging off
- Turn off power supplies
- Turn off gas flow
- Wait for the die temperature to drop below 200C when using a thermocouple or wait for the ram over temperature reading to drop below 100C if using the pyrometer.
- Save screen shot of Spec view software
    - Select Fn/F11 open word pad and paste image
    - Save to “My Documents” folder as experiment number
- Copy Log file and screen shot to USB thumb drive
    - Copy screen shot from “My Documents” folder
    - Copy log file from C:\SPS Data folder
- Open Roughing valve
- Allow system to pump down to below set point (5.0e-2)
- Close roughing valve
- Open Backfill valve
- Open the door. **Note**: parts may still be very hot and caution should be used before touching tooling.
- Use the Ram Enable pushbutton and Ram Jog switch to move the rams to an unloading position.

- Remove the punch and die assembly.

8) Administrative task.

- Complete the web information as appropriate.
- PID values are found in the iTools software under LP1_setup expanded folder.
    - Double click the PID folder and record the PB1,Ti1,PB2,Ti2,PB3,and Ti3 values.
    - Browse to the USB thumb drive and upload the log file and screen shot

9) System Shutdown

- Put graphite spacers and castle sections in the vacuum chamber
- Close chamber door
- Open roughing valve and allow system to pump down in order to store parts under vacuum
- Close roughing valve
- Shut off power supplies
- Shut off hydraulic pump
- Shut off vacuum pump
    - Open pump vent valve
- Shut off chiller
- Shut off system main and lock out
- Close computer widows and shut down
- Store computer in secure location.