# Operation Manual

## 5CM X 5CM ESC TEST FIXTURE KIT

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## PARTS INCLUDED IN TEST KIT

| Part Name | Description | Quantity |
|---|---|---|
| 5cm x 5cm ESC Cells | Cells included in the kit for testing. | 5 |
| 5cm x 5cm Alloy-X Manifolds | Test manifold. | 2 |
| 5cm x 5cm Cell Seal | Seals for the cell, seals cell to current plate. | 15 |
| 5cm x 5cm Manifold Seal | Seals for the manifold, seals manifold to current plate. | 15 |
| LNCCu Ink | Contact paste for the cathode, 100g pot. | 1 |
| NiNiO Ink | Contact paste for the anode, 100g pot. | 1 |
| Platinum Wire | Voltage sensing wire. | 1 m |
| Silver mesh cathode current collector | Silver cathode meshes, pre-cut and shaped. | 5 |
| Nickel Foam anode current collector | Nickel anode meshes, pre-cut and shaped. | 5 |
| Cersleev Insulation | Insulation for voltage sense and current wires. | 2 m |
| Braided Silver Wire | Current wire. | 1 m |
| 5cm x 5cm Current Plate | Current collection plate. | 2 |
| Never-Seez Can | Anti-Seize used with bolts on current plate. | 1 can |
| ¼"- 20 x 3/8" Bolts / Washers | Used to secure current wires to current plates. | 10 |

## REQUIRED TOOLS AND EQUIPMENT

| Equipment | Description | Quantity |
|---|---|---|
| Furnace | Appropriately sized furnace capable of 1000 °C maximum temperature. | 1 |
| Compression System | Used to compress seals. | 1 |
| Anode Humidifier (optional) and Condensate Collection | Used to predict OCV, collection of condensate to prevent blockages of exhaust. | 1 |
| Electronic Load | Used to collect performance data. 20A rating recommended for 5cm x 5cm cells. | 1 |
| AC Impedance Spectroscopy | Used to study electrode characteristics and resistances. | 1 |
| Gas Controls | Used to control anode and cathode gas compositions. 1 SLPM or greater maximum flow recommended. | 1 |
| Furnace Furniture and Insulation | Used to properly position cells and manifolds, insulate gas and current pass-throughs. | N/A |

## INTRODUCTION

The purpose of this operating manual is to provide in detail the steps required to prepare, assemble, and test 5cm x 5cm ESC cells. There are several auxiliary pieces of equipment that are needed to successfully test that are not included with this kit. This manual will make some assumptions on what that equipment is capable of, and how it will interface with the test kit.

**Please contact us if there are any unique setups or configurations that need to be considered before purchasing the testing kit.**

**The seals included in this kit must be compressed to seal effectively. The recommended amount of compression for the 5cm x 5cm test kit is 40 lbf (178 N) of compressive force evenly applied to the faces of the manifolds. Common methods to apply this force are:**

- **Use of blocks of high temperature steel as weights**
- **Pneumatic or hydraulic cylinders**
- **Screw or lever clamping systems**

**The use of a condensate collection system is recommended. This will prevent blockages in exhaust plumbing and allows for measurement of water generation rate, which could assist in data analysis.**

**The manifolds are supplied without fittings and with long straight tubes attached. This is to allow the user to choose fittings that will be compatible with their gas and exhaust infrastructure. The long tubes allow the user to bend or cut as needed to fit test furnaces and interface with gas controls.**

## SAFETY PRECAUTIONS

**There are no intrinsic hazards associated with the 5cm x 5cm test kit; however, safe operation of the kit requires the user to consider multiple sources of potential danger. There are three major hazards when operating the test kit.**

- **Explosive, flammable, and toxic gases – Be sure to connect exhaust lines to appropriate ventilation. Use flammable gases only in concentrations outside of their flammability or explosion limits unless there are appropriate measures in place to protect against explosion. The manifold set is NOT shipped in an oxygen clean state; use in pure oxygen is not recommended.**
- **Electrical shock – The manifold itself is not energized while testing; however, the current wires and voltage sense wires are. Use appropriate insulation and connections outside the furnace to prevent shorting. Ensure that the test kit does not contact the heating elements of the test furnace. This can energize the manifold and present a shock hazard.**
- **Hot surfaces – The furnace and any tubing or wires within will become extremely hot during operation. Use insulation, PPE, and guarding to prevent burns.**

## ASSEMBLY AND LOADING INSTRUCTIONS

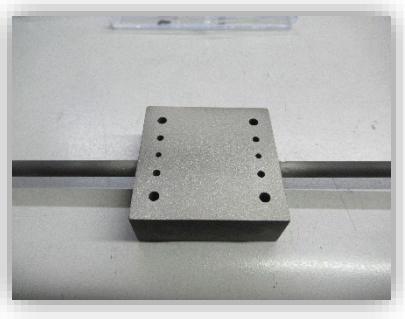
### Before Testing:

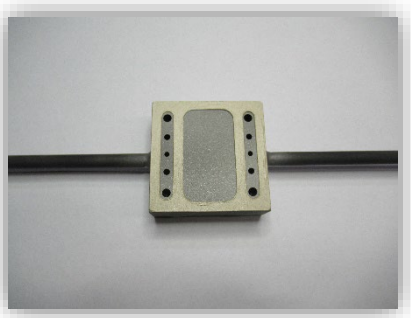
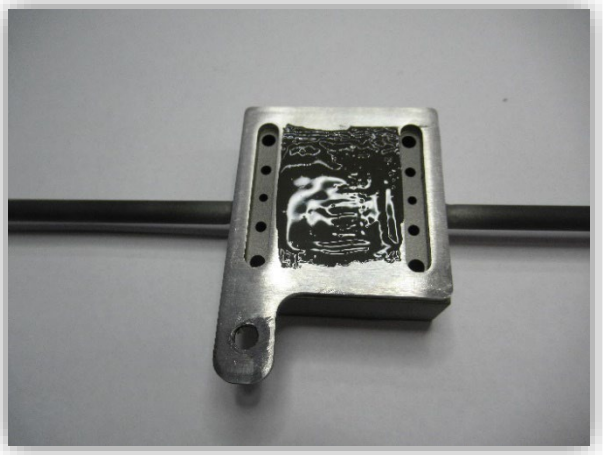
- **Before building a cell in the test fixture, test fit the manifolds in your furnace to see what bends, if any, are required for tubing, and where the voltage sense and current wires will exit. Choose what fittings and electrical connections you will use to control gas composition and collect data.**
- **This test fixture performs best when configured for co-flow, where the fuel and air inlets are on the same side of the manifold pair. However, you can test counter-flow by changing the inlet and outlet locations, or crossflow by rotating one manifold by 90 degrees.**
- **The manifolds do not require any heat treatment or conditioning prior to first use.**
- **You may use cyanoacrylate glue on the corners of the seals during assembly to prevent movement.**

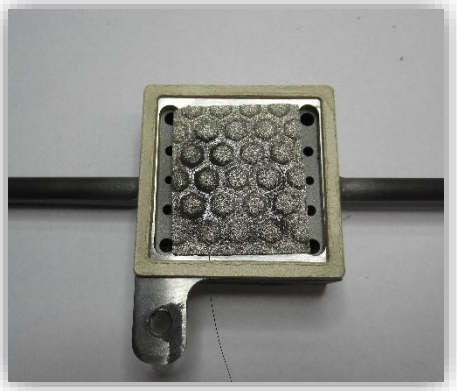
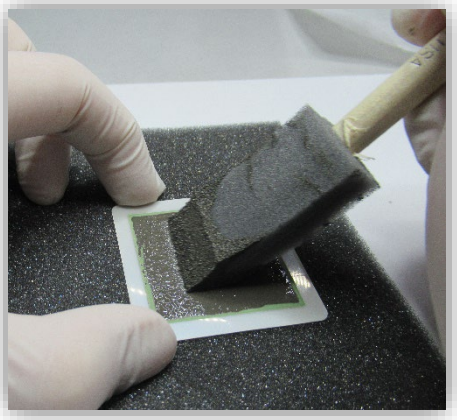
### After Testing:

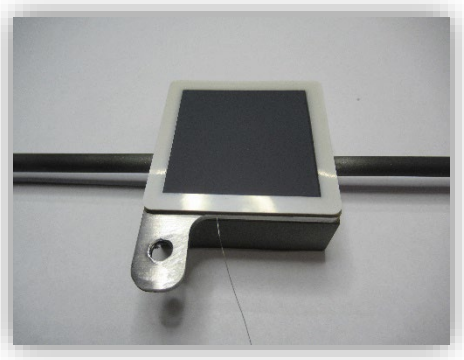
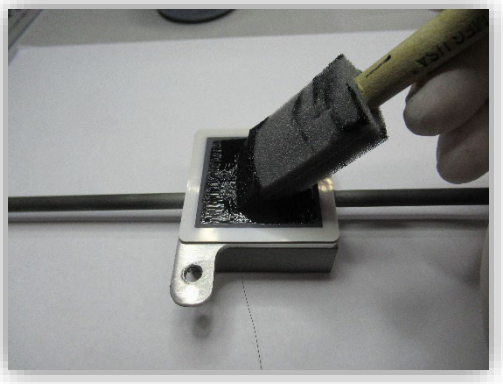
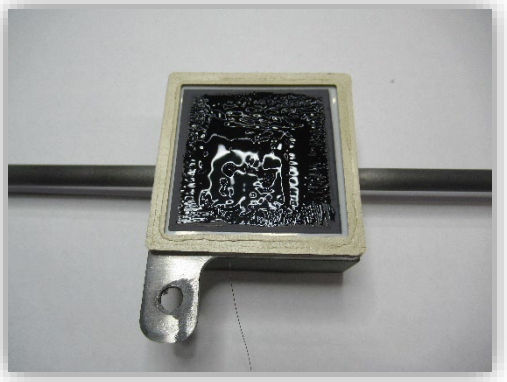
- **It is recommended that between tests all surfaces of the manifolds and current plates are sandblasted.**
- **Especially important are the seal areas and the current plate tab where the silver current wire makes contact.**

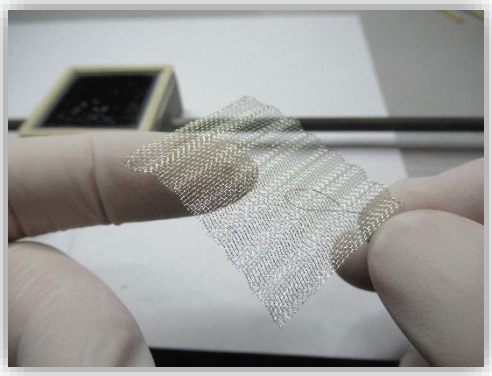
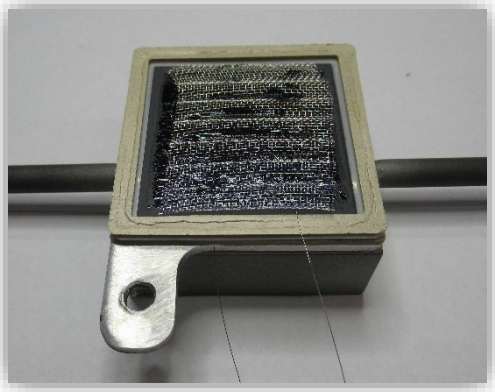
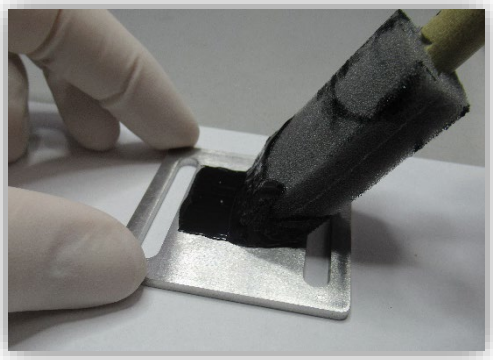
### Cell Assembly:

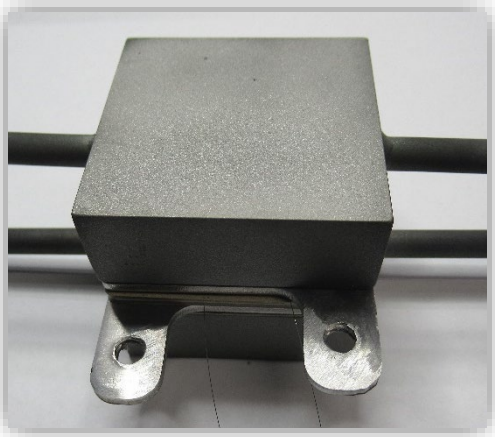
| Step | Description |
|---|---|
| 1. **Set Anode Manifold on Flat Stable Surface** | • You may apply cyanoacrylate glue to the corners of the manifold on this step.  |

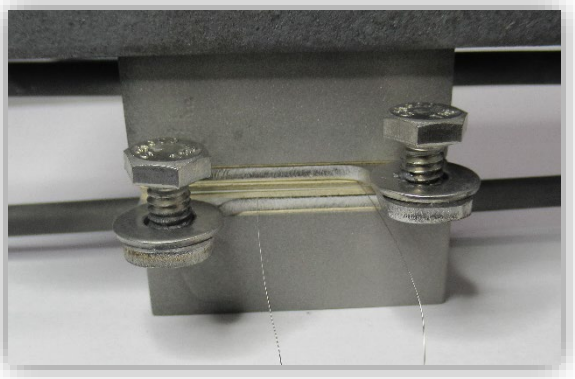
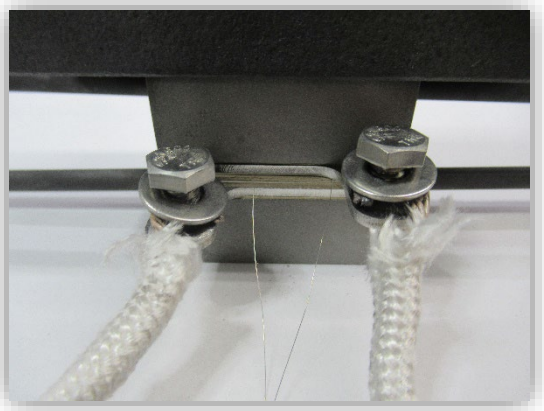
| | |
|---|---|
| **2. Place the Anode Manifold Seal on the Manifold** | • Be sure not to obstruct any of the gas ports.<br> |
| **3. Apply NiNiO Contact Paste** | • Apply approximately 0.5 grams of the provided nickel based ink to the surface of the current plate.<br>• Ensure the current tab is in the desired orientation.<br>• Be careful not to paint contact paste over the seal area.<br> |
| **4. Place the Anode Current Plate onto the Anode Manifold Seal** |  |

| | |
|---|---|
| **5. Place the Anode Voltage Sense Wire and Nickel Foam onto the Current Plate** | • Cut the anode voltage sense wire to an appropriate length.<br>• Place the anode voltage sense wire directly into the wet contact paste, and then place the Ni foam.<br>• Be sure to have the raised pattern of the nickel foam facing up.<br>• You may trim the foam to prevent any overlaps with the seal or gas ports.  |
| **6. Place the Anode Cell Seal** | • You may use cyanoacrylate on the corners of the seal on this step.  |
| **7. Apply Contact Paste to the cell Anode** | • Evenly apply approximately 0.5 grams of the nickel based contact paste to the anode of the cell.<br>• Verify the seal area of the cell does not have any contact paste applied.  |

| | | |
|---|---|---|
| **8. Place the Cell onto the Anode Cell Seal** | • Ensure the cell is aligned with the edges of the manifold and current plate. |  |
| **9. Apply LNCCu Contact Paste** | • Evenly apply 0.75 grams of the LNCCu contact paste to the cathode/air electrode.<br>• Verify the seal area of the cell does not have any contact paste applied. |  |
| **10. Place the Cathode Cell Seal onto the Cell** | • Ensure the seal is aligned with the edges of the manifold, current plate, and the cell.<br>• You may use cyanoacrylate on the corners of the seal on this step. |  |

| | | |
|---|---|---|
| **11. Attach Cathode Voltage Sense Wire to Cathode Current Collector** | • Make a small hook with the cathode voltage sense wire, then loop the hook through the silver mesh current collector. |  |
| **12. Place the Cathode Current Collector on the Cell** | • Place the silver mesh current collector onto the wet LNCCu contact paste. The cathode voltage sense wire should lie on top of the cathode cell seal. |  |
| **13. Apply LNCCu Contact Paste to the Cathode Current Plate.** | • Apply 0.5 grams of the provided LNCCu ink to the surface of the current plate.<br>• Ensure the current tab is in the desired orientation.<br>• Be careful to not paint contact paste over the seal area. |  |

| | | |
|---|---|---|
| **14. Place the Cathode Current Plate onto the Cathode Cell Seal** | • Ensure the current plate is aligned with the components below. |  |
| **15. Place the Cathode Manifold Seal onto the Cathode Current Plate.** | • You may use cyanoacrylate on the corners of the seal on this step. |  |
| **16. Place the Cathode Manifold onto the Cathode Manifold Seal** | • Ensure the manifold is aligned with the rest of the components. |  |

| | | |
|---|---|---|
| **17. Apply Anti-Seize to Threads of Bolt** | • Apply the compound evenly to the threads of the bolt. |  |
| **18. Place Current Wire Bolt.** | • Thread the bolts into the current plates. Make sure to add washers. **<u>You must apply anti-sieze only to the threads of the bolt.</u>** |  |
| **19. Attach Current Wires** | • Secure the manifold and cell assembly with either a weight or a clamp, and attach the silver current wires to the current plates using the bolts. |  |

| | |
|---|---|
| **20. Tighten Current Wires** | • Using a 7/16" wrench, tighten the bolts to secure the current wires.<br> |
| **21. Transfer Assembly to Furnace** | • Ensure all seals, plates and cells are aligned in the furnace before applying compression.<br>• You may insulate the voltage sense wires with Cersleev once the manifold is in place in the furnace.<br> |
| **22. Apply Compression** | • Apply 40 lbf ( 178N ) to the assembly. |
| **23. Connect Gas Lines, Voltage Sense, and Current Wires.** | • Attach the gas inlets to your gas control system. Connect exhausts. Connect the voltage sense and anode current wire to your electronic load.<br>• Insulate all openings to furnace, taking care to prevent shorts between the sense wires or current wires.<br>• **You can check for continuity between the voltage sense wires or current wires while the furnace is cool to check for shorts.**<br>• The system is now ready for cell conditioning. |

## CELL CONDITIONING

**Before heating up the test assembly, check that the cathode current wire is not connected to the electronic load. Follow the recipe below to heat-up, condition, and reduce the cell. Conditioning is considered the heating of the cell with nitrogen on the anode and air on the cathode. Reduction begins once the furnace has reached 850 °C, and the hydrogen concentration slowly begins to increase. The recommended ramp rate for this kit is 1°C/min.**

| Cell Conditioning / Reduction | | | | | |
|---|---|---|---|---|---|
| Furnace and Dwell Times | | | Oxygen Electrode | Fuel Electrode | |
| Step | Time [hh:mm:ss] | Temperature [°C] | Air Flow [SLPM] | Nitrogen Flow [SLPM] | Hydrogen Flow [SLPM] |
| 1 | 14:00:00 | 0 →850 | 0.150 | 0.150 | 0.000 |
| 2 | 0:30:00 | 850 | 0.250 | 0.150 | 0.050 |
| 3 | 0:30:00 | 850 | 0.500 | 0.150 | 0.075 |
| 4 | 0:30:00 | 850 | 0.500 | 0.100 | 0.125 |
| 5 | 0:30:00 | 850 | 0.625 | 0.050 | 0.175 |
| 6 | 0:30:00 | 850 | 0.750 | 0.000 | 0.225 |

**The cell is considered fully reduced when the OCV has stabilized. The cell is now ready for testing, and you may connect the cathode current wire to the electronic load.**