fuelcellmaterials.com
PERFORMANCE AND QUALITY DELIVERED
404 Enterprise Drive
Lewis Center, Ohio 43035

5 cm x 5 cm Anode Supported Cell Test Fixture Operation Manual

Included in 5 cm x 5 cm Test Fixture Kit

- ☐ 1 - pair manifolds
- ☐ 5 - 5cm x 5cm Anode supported cells
- ☐ 15 - seals cut to 5cm x 5cm test fixture specification
- ☐ 100 grams - LSM cathode ink
- ☐ 50 grams - nickel anode ink
- ☐ 475 milliliters high temperature sealing paste
- ☐ 1 meter - platinum wire
- ☐ 5 - silver cathode meshes, pre-cut and shaped
- ☐ 5 - nickel anode meshes, pre-cut and shaped
- ☐ 1 – operation manual
- ☐ 1 meter - CerSleev (insulating ceramic cloth tubes)
- ☐ 10 grams - silver contact paste
- ☐ 1 meter - copper braided leads

Required tools and Equipment

- ☐ One furnace with a maximum temperature of 1200°C
- ☐ Compression system
- ☐ Anode humidity (optional) and condensation collection systems
- ☐ Electronic load
- ☐ AC Impedance Spectroscopy (optional)
- ☐ Gas control
- ☐ Kiln furniture and insulation
- ☐ Clamps to hold current leads to manifold tubes

Introduction

Below are step by step instructions on proper assembly of the 5cm x 5cm Test Fixture Kit for testing.

The seals in the test kit are compression seals; therefore a compression system must be used to get accurate results. This can be done a number of different ways; the two most common are to apply a series of weights (e.g. blocks of high temperature steel) onto the top of the manifold or to use a clamping mechanism. A pressure of 5 PSI should be targeted with either method in order to give you the compression needed to have the seals work properly. Careful preparation of the furnace and insulation is absolutely critical to loading the test fixture without damaging the cell. The instructions below assume a top-opening furnace is used, though they do not give explicit preparation instructions. Adjust your methodology accordingly to your own furnace.

The manifolds are provided without fittings or electrical connections so that they can be cut and/or bent to the appropriate dimensions to fit your furnace and periphery testing equipment. In order to achieve the best performance we recommend following the manifold

preparation steps below. Also recommended is an anode outlet condenser to measure water generated during your experiments.

SAFETY NOTES

While there are no intrinsic hazards associated with the 5cm x 5cm Test Fixture Kit, safe operation requires that the user considers potential dangers and unexpected situations that can be caused by the gases (flammable, oxidizing and inert), pressures, temperatures, electricity or the cell itself. Further, the user must ensure that the fixture is used according to local safety regulations. We recommend use of safety glasses when working with the fixture at all times.

Potential hazards to consider are:
Explosive, toxic and oxidizing gases

- For toxic or flammable gases, be sure to connect the fixture outlets to a suitable ventilation/hood system, and have appropriate alarm and shut-off systems in operation in case of leakage or failing ventilation.
- Use flammable gases only in concentrations below or above the explosion limits unless appropriate actions are taken to prevent damage in case of explosion and approval to use such gases is given by your local authority.
- Familiarize yourself with the symptoms of toxic gas exposure and actions to take in case of accidental exposure.
- Under certain conditions, pure oxygen may spontaneously ignite oxidisable materials such as certain metals and organics, and the subsequent reaction may proceed explosively. Thus, keep the fixture clean and free of foreign materials.

Electrical shock
Do not connect potentially dangerous high voltages to the electrical connections of the fixture. If possible, use furnaces with low or floating voltages. Ensure that the fixture cannot be brought into contact with the heating element. Some kind of insulation (e.g. ceramic tube) between the heating element and the fixture is preferred. If necessary, install extra physical hindrances or insulators to prevent such accidental contact. As with electrical equipment in general, avoid touching a potentially voltage-carrying object with one hand and a grounded object with the other. If applicable, ask your local electrical safety personnel to approve your installation of a furnace and other equipment.

Do not touch any part of the fixture during heat-up, conditioning, experimentation or cool-down to prevent any possibility of electrical shock.

Hot surfaces
Do not touch any part of the fixture during heat-up, conditioning, experimentation or cool-down to prevent any possibility of burns. The gas feed tubes of the fixture will likely be quite hot. Furthermore, the fixture may retain heat for a long time after the experiment. The user should exercise caution and utilize appropriate protective equipment whenever handling the fixture after an experiment.

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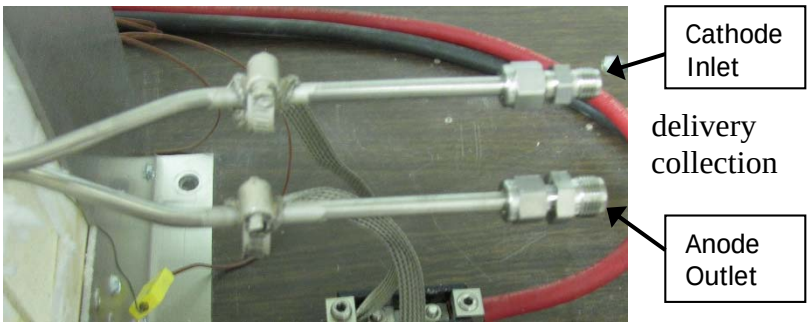
Manifold Preparation

1. Place the fresh manifolds in your furnace to determine how they align with the furnace, gas system and current system. Cut and/or bend the inlet and outlet tubes as necessary to provide the best alignment. Apply fittings to the anode and cathode inlet tubes, anode exhaust tube and cathode exhaust tube (optional if using air). This test fixture performs best in a cross flow configuration – the anode and cathode gas enter the manifold on opposite sides, crossing each other as they traverse the cell. To achieve this, the tubing should be set up so that the cathode inlet is on the same side as the anode exhaust and vice versa (Figure 1).



2. Measure the distance from your manifold tubes to your current collection system (Figure 2). Cut the included copper braided leads into 2 pieces, each a slightly longer length than the measured distance.

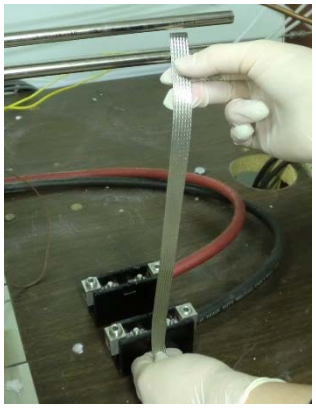


Figure 2

3. In order to ensure good electrical contact, apply a layer of the included silver ink to the manifold tube where the lead will make contact (Figure 3). Wrap the lead around the tube (Figure 4) and apply a clamp to hold the lead to the tube (we recommend hose clamps) – Figure 5. This should be repeated on one tube of the other manifold, so that each manifold has one current lead attached.

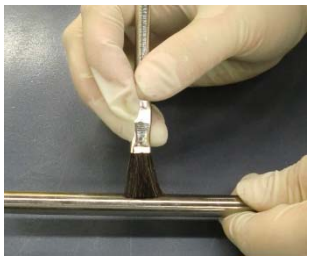


Figure 3


Figure 4

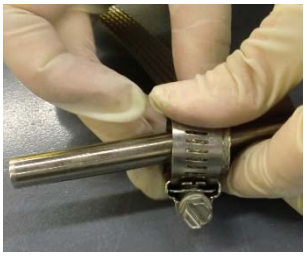


Figure 5

4. Before first use with a cell, the manifolds should be fired to the intended test operation temperature for approximately two hours in order to burn out any residues from manufacturing and to create a protective oxide coating on the cathode manifold. Figure 6 shows fully prepared and fired manifolds.

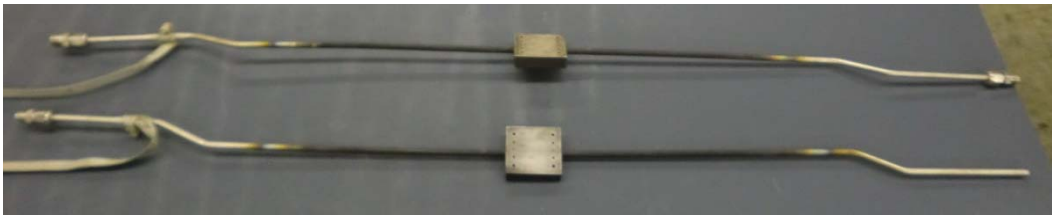


Figure 6

Cell Loading

1. Place the anode manifold on a flat, stable surface. Apply small drops of cyanoacrylate "superglue" to all four corners and place a seal on the manifold. (Figure 7 & 8)

Figure 7

Figure 8

2. Apply approximately 0.5 grams of the provided nickel ink on the surface of the anode manifold (Figure 9). Do not apply ink to the gas flow ports.

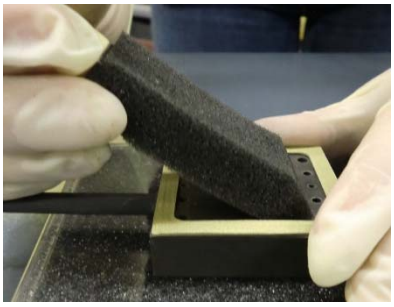


Figure 9

3. Place the nickel mesh on the active area of the manifold so that the raised pattern is facing up. Make sure the nickel mesh does not overlap the seal or cover the gas flow ports (Figure 10). Trim the nickel mesh to a slightly smaller size if necessary to prevent overlap.

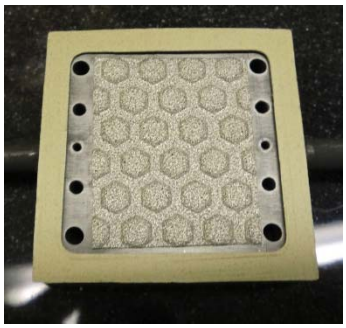


Figure 10

4. Use approximately 0.5 grams of nickel ink to evenly cover the anode active area of the fuel cell with a thin layer of ink (Figure 11).

Figure 11

5. Measure and cut the platinum voltage wire into two wires of sufficient length to extend from inside your furnace to your voltage monitoring connection. Bend a small hook into one platinum voltage wire and place it in the middle of the Ni mesh on the anode manifold (Figure 12). Typically it is difficult to secure the wire in place, so Step 6 should be performed while holding the wire from Step 5 in place with one hand.

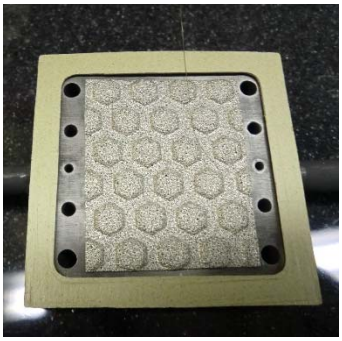


Figure 12

6. Carefully align the cell and place it anode side down against the voltage wire and nickel mesh (Figure 13).

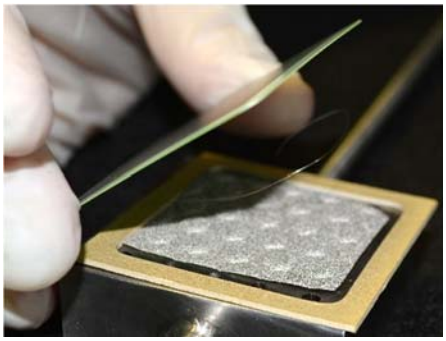


Figure 13

7. Lightly holding the cell in place, evenly apply approximately 0.75 grams of LSM ink to the cathode active area (Figure 14).

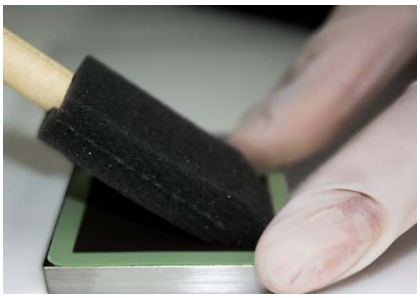


Figure 14

8. Bend a small hook into the remaining platinum voltage wire and place it in the middle of the painted cathode active area (Figure 15). Ensure that you do not place the cathode voltage wire directly on top of the anode voltage wire – these must not be in contact during testing.

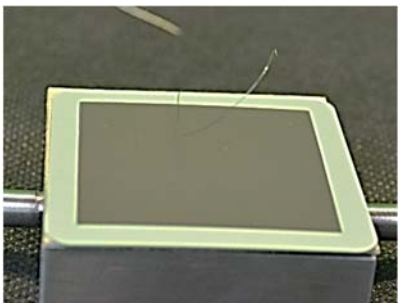


Figure 15

9. Place the silver mesh onto the cathode active area so that the channels are parallel to the manifold tubes (Figure 16).

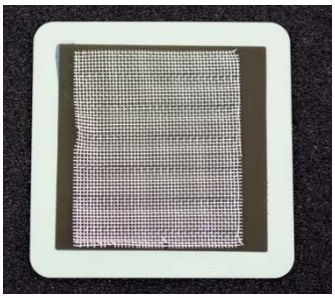


Figure 16

10. Place the remaining seal on the cell so that it is in alignment with the edge of the cell and manifold (Figure 17). Make sure the seal does not overlap the silver mesh; trim the silver mesh to a slightly smaller size if necessary to prevent overlap.

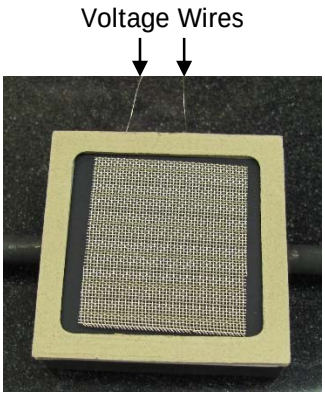


Figure 17

11. Apply approximately 0.5 grams of LSM ink in a thin even coat on the active area of the cathode manifold (Figure 18). Do not apply ink to the gas ports.

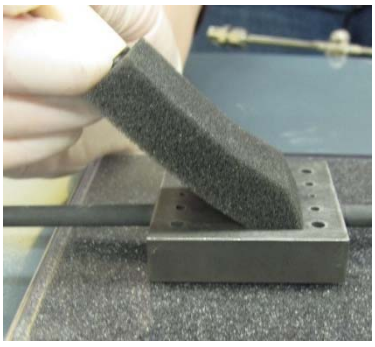


Figure 18

12. Place the cathode manifold on top of the cell assembly. Apply the high temperature sealing paste around the perimeter of the cell and manifold. Allow the paste to cure at room temperature, or cure in-situ within the test furnace. This paste will seal the porosity of the anode cell and help form and gas tight seal. (Figure 19).

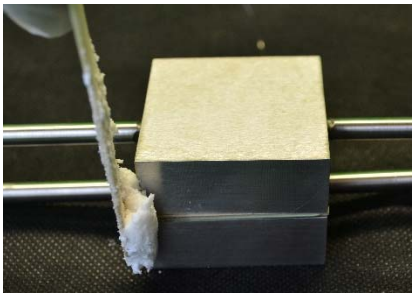


Figure 19

13. Carefully place the manifold assembly with cell into the furnace (Figure 20).

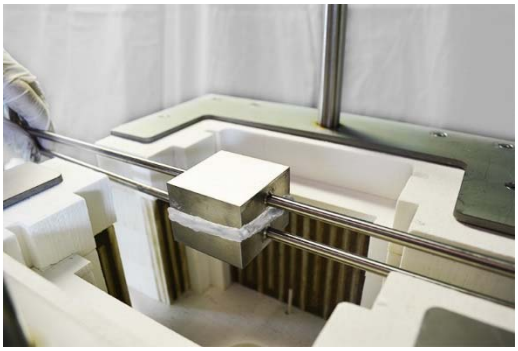


Figure 20

14. Place a small amount of flexible insulation between the anode tubes and cathode tubes of the manifold in order to prevent the tubes from touching and isolate them electrically. (Figure 21).

Figure 21

15. If possible, apply your compression system to the test fixture to ensure that it does not shift during the remaining steps.

16. Place insulating ceramic cloth tubes (CerSleev) over the anode and cathode voltage wires to isolate them from the furnace and one another (Figures 22 and 23). To make it easier to run the wires through the cloth tubes, any solid tube can be run through the cloth first, the wire fed through and the solid tube removed.

Figure 22

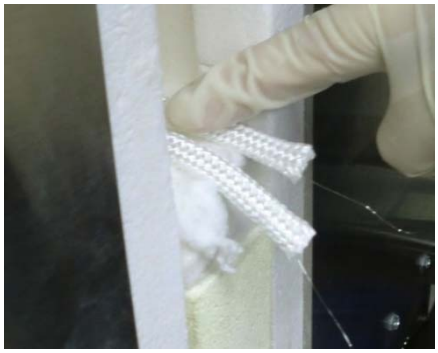


Figure 23

17. Make the appropriate gas inlet and current cable connections (Figure 24). Usually the outlet tubes are left unconnected during heat-up to allow easy clean-up of any binders that condense at the end of the tubes.

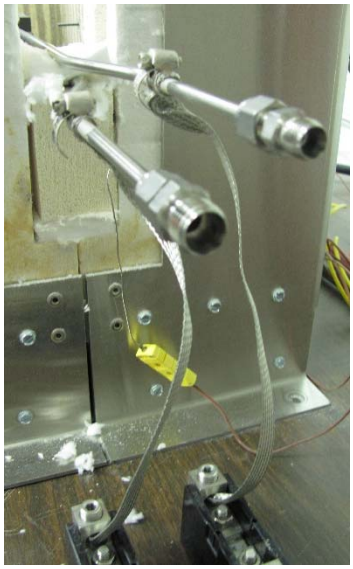


Figure 24

18. Connect the anode and cathode voltage wires to appropriate clips and secure in place to prevent pulling or otherwise stressing the connections.

19. The set-up is now complete.

Cell Conditioning

Note: The following steps are recommendations only. The exact settings needed may vary depending on your set-up and testing goals.

1. Unplug the cathode current cable from the control box and turn the load box component off to ensure no current is passing through the system during heat-up.

2. Program the furnace to heat up to 850°C in 14 hours (~1°C/min) and hold at 850°C.

3. Increase the anode and cathode flows to appropriate desired flow rates for heat-up.
   - Cathode: air @ 150 sccm (ml/min)
   - Anode: dry N_2 @ 150 sccm

4. Start the furnace program.

5. Allow the cell and furnace to heat overnight.

Cell Reduction

Note: The following steps are recommendations only. The exact settings needed may vary depending on your set-up and testing goals.

1. Switch on the load box component of the control box and record the open circuit voltage (OCV).

2. Before proceeding, you may wish to clean away any binder that may have condensed at the ends of the metal outlet tubes.

3. Connect the anode outlet tube to the anode gas humidity condenser (if desired) and/or safe anode exhaust system.

4. Maintaining 150 sccm N_2 on the anode, increase the H_2 flow to 50 sccm.

5. Increase the cathode air flow to 250 sccm.

6. Allow the system to sit for 20-30 minutes or until the OCV begins to stabilize, whichever is longer.

7. Increase the H_2 to 75 sccm and allow the system to sit for another 10 minutes.

8. Transition the anode feed stream to 225 sccm H_2 and 0 sccm N_2 in 50 sccm increments allowing the cell to sit at each condition for 5-10 minutes (or until the OCV begins to stabilize, whichever is longer).

9. Concurrently transition the air flow up to 750 sccm in even increments.

10. Once the desired gas flows of 225 sccm H_2 and 0 sccm N_2 on the anode and 750 sccm air on the cathode are reached, allow the system to sit for 15 - 30 minutes to assure steady state.

11. Connect the cathode current cable at the control box.

SOFC Manifold/Fixture is ready for testing