**Purpose:**

The purpose of this procedure is to describe the process used to oxidize parts used in the assembly of sensors having Bismuth Titanate crystals. This process is often referred to as “Blue-Oxide”.

**Responsibilities:**

HCD Department engineering / management is responsible for maintaining this procedure. HCD Department technicians are responsible for carrying out this procedure.

**Associated Documents: ISO 9001, QAM, QSM, AS9100, TA1051**

**1.0 General Practice**

Certain high temperature sensors use bismuth titanate as a sensing material. It has been found that bismuth titanate loses its insulation resistance in a reducing atmosphere (removal of oxygen). Since our sensors are in a hermetic metallic housing, at high temperatures, the metal parts will oxidize. As this process happens, a reducing atmosphere inside the sensor is created, causing the bismuth titanate to cease functioning.

To help prevent the depletion of oxygen it is necessary to pre-oxidize the parts. However, we do not want to put such a heavy oxide that we can no longer weld the sensor. Also, a heavy oxide can create too much scaling on critical surfaces leading to performance issues of the sensor. So, it is critical that we create an oxide heavy enough as to not steal oxygen from inside the sensor, but not too heavy to prevent welding/performance issues. The procedure outlined below (“blue-oxide”) has been found to give us the best chance for success.

**2.0 Safety Precautions**

This procedure requires the use of high temperature furnace, operating at approximately 1200ºF. During this procedure, the operator is required to load parts into the preheated furnace. **Extreme caution must be used during this process. Only trained operators are allowed to perform this operation, and appropriate safety equipment must be worn. High temperature/heat resistant gloves are required whenever inserting or removing parts from the oven. Whenever opening the oven at temperatures above 900°C, the shaded visor must be worn to protect your eyes and skin. The provided tools must be used when loading and unloading the furnace.**

Methanol is used for cleaning in this procedure. Methanol is flammable and must be handled with care and should be stored in the proper safety cabinet for flammable liquids when not in use. Keep away from heat, hot surfaces and sparks. Label all containers appropriately, and dispose of used chemicals in the appropriate chemical waste container.

**3.0 Equipment and Materials**

Sentry Furnace

Tweezers

Finger cots or powder free gloves

Parts per BOM

Furnace loading tool (“pitchfork”)

High temperature Firing Trays (48003-01 or similar)

Heat Resistant Gloves

Face shield/visor

Ultrasonic cleaner

Methanol

Beakers

Petri Dish

Crystallizing Dish

Metal Basket

High Temp drying oven (CA 3157)

Hand held infrared thermometer Omega OS-DT8855W or equivalent

**4.0 Pre-Clean Electrodes and Diaphragms**

**\*\*All ultrasonic cleaning must be done in a well ventilated area.\*\***

## Note: **Parts are not to be touched after cleaning** to preclude contamination. Handle with tweezers, clean gloves or finger cots and place in clean containers that will protect against damage during transfer and storage. Label as “cleaned [DATE]” if parts are not to be used immediately.

4.1 Gently place each electrode or diaphragm in a small clean beaker.

Note: Electrodes and diaphragms should be cleaned individually (one per beaker).

4.2 Put on PPE including: chemical goggles and nitrile gloves.

4.3 Add enough fresh Lenium to each beaker to cover the part(s) at least a ¼”.  
Note: There needs to be enough Lenium in the beaker so that it does not tip over when placed in the Ultrasonic.

4.4 Make sure there is DI water about ½” above the base and insert tray of the Ultrasonic.

4.5 Place beakers in the insert tray of the Ultrasonic.

4.6 Turn the Ultrasonic on and clean parts at room temperature with sonics for 5 minutes. If necessary, see HC1018 for more instruction on how to operate the Ultrasonic.

4.7 Remove beakers from the insert tray, making sure not to drip water into the other beakers.

4.8 Drain beaker(s) into a lenium waste container. Use a strainer if necessary.

4.9 Add Methanol to each beaker to cover the part(s) by at least a ¼”.

Note: There needs to be enough Methanol in the beaker so that it does not tip over when placed in the Ultrasonic.

Note: Methanol should be poured in a well ventilated area.

Note: When Methanol is not being used, make sure to store the bottle in the flammable liquid safety storage cabinet. Do not leave the bottle out on the benchtop.

4.10 Place beakers in the insert tray of the Ultrasonic.

4.11 Cover the ultrasonic cleaner with the lid.

NOTE: The cover/lid must be on whenever there are beakers of Methanol in the ultrasonic cleaner

4.12 Turn on the Ultrasonic and clean the parts at room temperature with sonics for 5 minutes. If necessary, see HC1018 for more instruction on how to operate the Ultrasonic.

4.13 Remove beakers from the insert tray, making sure not to drip water into the other beakers.

4.14 Drain beakers into a Methanol waste container using a strainer and funnel if needed.

NOTE: Safety disposal can and/or funnel must be grounded before pouring Methanol waste.

NOTE: Do not leave funnel in the disposal can. The lid of the disposal can MUST be closed when it is not in use.

4.15 Dry parts in 110C ± 10C drying oven for twenty (20) minutes minimum.

**5.0 Pre-Clean MS Parts**

**\*\*All ultrasonic cleaning must be done in a well ventilated area.\*\***

## Note: Parts are not to be touched after cleaning to preclude contamination. Handle with tweezers, clean gloves or finger cots and place in clean containers that will protect against damage during transfer and storage. Label as “cleaned [DATE]” if parts are not to be used immediately.

5.1 Gently place parts into a metal basket.

5.2 Put on PPE including: safety goggles and nitrile gloves.

5.3 Ultrasonic degrease parts per HC1001, 5 minutes with ultrasonics.

5.4 Place parts in a clean petri or crystallizing dish with a lid.

5.5 Gently place parts into clean beaker(s) or crystallizing dish(es). Insure units with delicate components are appropriately spaced to avoid damage.

5.6 Add Methanol to the beaker(s)/dish(es) to cover all the parts.   
Note: Methanol should be poured in a well ventilated area.

Note: When Methanol is not being used, make sure to store the bottle in the flammable liquid safety storage cabinet. Do not leave the bottle out on the benchtop.

5.7 Place beaker(s)/dish(es) in the insert tray of the Ultrasonic.

5.8 Cover the ultrasonic cleaner with the lid.

NOTE: The cover/lid must be on whenever there are beakers of Methanol in the ultrasonic cleaner

5.9 Turn on the Ultrasonic and clean the parts at room temperature with sonics for 5 minutes. If necessary, see HC1018 for more instruction on how to operate the Ultrasonic.

5.10 Lift the beaker(s)/dish(es) out of the insert tray.

5.11 Drain beaker/dish into a Methanol waste container using a strainer and a funnel if needed.

NOTE: Safety disposal can and/or funnel must be grounded before pouring Methanol waste.

NOTE: Do not leave funnel in the disposal can. The lid of the disposal can MUST be closed when it is not in use.

5.12 Dry parts in 110C ± 10C drying oven for twenty (20) minutes minimum.

**6.0 Load Parts**

6.1 Remove parts from the drying oven.

6.2 Load parts onto Firing Trays.

NOTE: Do not handle the tooling or parts with bare hands. Always wear finger cots or powder free latex gloves, and use tweezers to handle small parts.

6.3 Multiple parts can be loaded if you insure that flat surfaces are not touching other flat surfaces. Edges (line contacts) and corners (point contacts) touching are acceptable.

6.4 Parts such as diaphragms and electrodes will have to be laid flat, but must not overlap each other or the desired oxide will not result on these surfaces.

**7.0 Oxidize Parts**

7.1 Program the Sentry furnace to 1200°F or 649°C. Allow the furnace to stabilize at temperature for 1 hour minimum. This may and should be done ahead of time.

\*\* **NOTE: The following steps involve opening the door of a very hot furnace. Use extreme caution and wear the required personal protective equipment. \*\***

7.2 Set the timer. Oxidation time 2:45 to 3 hours.

7.3 Put on all necessary PPE including heat resistant gloves.

7.4 Insert the “pitchfork” tool under the firing tray(s) loaded with parts in 6.2.

7.5 Open the furnace door about 6 inches. Using the pitchfork, load the firing tray(s) into the furnace, carefully remove the pitchfork tool, and close the furnace door.

Note: No more than four (4) trays of heat treat or “blue oxide” parts may be placed in the furnace at one time.

7.6 Set the pitchfork on the heat-resistant surface. Be careful, the end of the pitchfork is very hot. Remove the gloves.

7.7 Start the timer.

7.8 When the timer times out, put on the gloves. Pick up the pitchfork.

7.9 Open the furnace door about 6 inches. Carefully insert the pitchfork under the firing tray(s) in the furnace, and lift and remove the tray(s) from the furnace. Set the hot tray onto the heat-resistant platform next to the oven, remove the pitchfork from the tray(s), and set aside. Close the furnace door. Remove the gloves, and set aside.

7.10 Allow the firing tray and assemblies to cool for at least one hour or until the tray and parts/tooling reach 30oC +/- 5oC. The temperature of parts can be read using the handheld infrared thermometer.

**NOTE: Do not attempt to handle or move the tray for at least one hour. Even after the color changes from orange to black, the tray can be extremely hot and cause serious burns.**

7.11 Once cool, remove the parts from the firing tray using tweezers or clean gloves/finger cots. Place parts into their appropriate packaging (plastic trays).

7.12 Repeat steps 6.2 through 7.11 until all the parts on the job are oxidized.