LASER INTERFEROMETER GRAVITATIONAL WAVE OBSERVATORY

LIGO Laboratory / LIGO Scientific Collaboration

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Preparation of an end or input test mass (ETM/ITM) (Hydroxide-Catalysis Bonding of ears)

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1 Introduction

1.1 Purpose and Scope

This document defines the procedure for preparing an ETM/ITM test mass (D080658-v3 and D080657-v3) for installation into the quadruple suspension structure. The procedure consists of hydroxide catalysis bonding on a fused silica 'ear' (suspension element according to D080751-v1) to each of the surfaces S3 and S4 in a well-defined location and re-storing the mass. General hydroxide-catalysis bonding procedures are detailed in E050228. The procedure assumes the mirror substrate has been fully prepared with HR and AR coatings applied to respectively the front and the back surface of the substrate. It includes mirror handling with the ergo-arm during these procedures. Mirror handling procedures with the ergo-arm are detailed in T1000082.

It assumes that the mass has been stored in a COC ETM storage container or 'cake-tin' (D0902146). Both coated surfaces have First Contact films already applied to them. The procedure starts from the point of removing the mass from the 'cake-tin' using the ergo arm. It finishes with the 4-week curing period needed to cure the ears. The test mass is stored back into the cake-tin container during this curing period.

The procedure does not include cleaning and/or baking procedure prior to inserting the mass into the structure. On the date this update was written this was not considered as part of the procedure for the silica masses.

The document starts with giving an overview of the required lab-settings and tooling followed by giving the relevant documentation.

It then discusses the detailed steps of the procedure including location, timing, number of personnel needed and tooling needed.

1.2 Required lab-settings

The surfaces must be free of particles, thus, the bonding must take place under a Class 100 laminar flow bench or in a Class 100 clean-room.

Operator must be dressed in clean room attire: overall, boots, head cover, facemask, and approved cleanroom gloves are required.

This clean-room has a sink with running DI water with a resistance of 18 MOhm. It also has two tables of normal height (between 70 and 75 cm).

1.3 Equipment and Materials

- Filtered dry nitrogen
- (De-)Ionizing gun
- High intensity light source (ideally handheld battery supported)
- DI water 18 MΩ resistance
- Pipettor with tip ejector - variable volume, 2-20 µl, Eppendorf 2000 - (VWR Cat. No. 53511-588)

- Microcentrifuge tubes - Eppendorf, 1.5 ml - (VWR Cat. No. 20901-551)
- VWR® MiniFuge Microcentrifuge - 120V, 50/60Hz - (VWR Cat. No. 93000-196)
- Microcentrifuge tubes storing rack - (VWR Catalog No. 20901-675)
- Centrifuge tubes - polypropylene, graduated, 15 ml – (VWR Cat. No. 21008-103)
- Centrifuge tube's rack – (VWR cat. No. 21008-485)
- Medical Filter: Whatman Filter Uniprep 0.2UM PK50 UN113ENYL Filter
- Eppendorf* epTIPS* Pipette Tips – Sterile PCR Clean Filter Tips, 2-20 μl, 10 Racks of 96 Tips – (VWR Cat. No. 47745-092)
- Alpha 10 wipes - case – (VWR Cat. No. TWTX1010)
- Gloves - VWR Certi-Clean Class 100 Latex Gloves or Accu Tech Ultra Clean 91300 Gloves.
- Methanol – Spectroscopic grade
- Acetone – Spectroscopic grade
- Sodium bicarbonate (= Bicarbonate of soda)
- Cerium oxide polishing compound
- Micro 90® detergent (International Products Corporation)
- Sodium Silicate Solution – from Sigma-Aldrich 338443-1L (~10.6% NaOH, ~26.5% SiO_2 by weight)
- Ultrasonic cleaner - BRANSON 8510
- 2x V-block - D1001685
- Bonding jig - D1001592
- Metric slip gauges
- Allen key for #2-56 socket-head cap screw
- Digital calipers – with metric setting
- UHV aluminium foil
- Glass petridishes
- Magnifying glass
- First contact
- Ergo-arm

1.4 References

Design documentation 'glass' essentials

D080657	ALIGO COC ITM SUBSTRATE
D080658	ALIGO COC ETM SUBSTRATE
D080751	ETM ITM quad production ear
D0902455	ETM bonded assembly
D0902456	ITM bonded assembly
Design documentation of the alignment jigs	
D0901592	ASSEMBLY DRAWING – NP-type bonding jig
D0901591	Baseplate – NP-type bonding jig
D1000128	Penultimate mass prism holder
Measurement reports on 'glass' essentials	
Q10xxyy	Relevant test mass control measurements
Q10xxxx	Relevant ear control measurements of ears
Back ground documents	
E050228	(Specification) Silicate Bonding Procedure
T0900402	Enhanced LIGO core optic drag wipe cleaning procedure
E0900394	aLIGO Optic Container Shipping Procedure
E1000079	Advanced LIGO First Contact procedure for large optics
T1000114	ALIGO NP-type: - Report on Ear Bonding at LASTI 27th August – 31st August
T0900447	Ear fabrication readiness review
M080134	ETM/ITM and BS/FM pitch frequencies and d-values
T1000534	Ergo-arm users manual
D0902146	ASSEMBLY, ETM, COC OPTIC CONTAINER, ADVANCED LIGO
E1000079	First Contact Application and Removal Procedure
F1000006	Bond quality monitoring form
E1000265	Jig-settings calculation sheet

1.5 Version history

04/08/10: Requested DCC number for the document.

08/04/10: Release v1 onto DCC.

18/02/2011: Release v2 onto DCC.

2 Main procedure

2.1 Set jig for the relevant side (“S3” or “S4”) of the mass

Step	What	Where	Time	People	Tools
1	Select the mass, which side (surface “S3” or “S4”), ear and bonding jig that will be used for the bonding. Calculate the required settings for D_{slider} and D_{screw1} for the bonding jig using the (E1000256) and copy onto clean-room paper	In office	30 min	2 (one calculator, one checker)	Quality control documentation of mass, ear and bonding jig.
2	Set D_{screw} on the left side of the jig (side 1 on the assembly drawing) for bonding onto surface “S3” or the right side of the jig (side 2 on the assembly drawing) for bonding onto surface “S4”	in bonding cleanroom	5 min	1	Bonding jig assy, slip gauges of the right thickness
3	Set D_{slider} on both sliders of the jig	in bonding cleanroom	5 min	1	Bonding jig assy, calipers, Allen key for #2-56 cap screw
			40 min		

2.2 Set-up mass and ear for cleaning

Step	What	Where	Time	People	Tools
4	Take the mass out of its ‘cake-tin’ and place onto the V-block on the washing table/sink. This is done using the ergo-arm. Follow instructions for cake-tin. **The bonding sides are placed vertically to minimize risk of rinsing water and methanol over the coated surfaces of the mass and to give easier access.** **Rinsing methanol over the surfaces coated with first contact should be avoided. The barrel and bonding flats should be wiped with acetone near coated surfaces for this reason. The bonding area should NOT be wiped with acetone. It HAS TO be wiped with methanol.**	in bonding cleanroom	15 min	2	Ergo-arm (T1000082), mass in ‘cake-tin’ with tooling to open,V-block, isopropanol, optical wipes

Step	What	Where	Time	People	Tools
5	Take the ear out of its packaging and place in a petridish on an optical cloth	in bonding cleanroom	2 min	1	Ear, petridish, optical wipe
6	Change gloves	in bonding cleanroom	1 min	2	Gloves
			18 min		

2.3 Prepare bonding solution

Step	What	Where	Time	People	Tools
7	Pour 2 ml of sodium silicate solution and 12 ml of DI water into a 15 ml centrifuge tube, close the lid, and shake thoroughly for 30 seconds (this gives a 1:6 volumetric ratio of sodium silicate solution to DI water)	in bonding cleanroom	1 min	1	15 ml centrifuge tube, sodium silicate solution, DI water
8	Transfer the solution into 3 1.5 ml centrifuge tubes. Take care you don't hover with any gear above the tubes at any time. Then close lids. Transfer to centrifuge and let them spin for 30 seconds.	in bonding cleanroom	1 min	1	Rack for 1.5 ml centrifuge tubes, 1.5 ml centrifuge tubes, centrifuge
9	Open up two of three centrifuge tubes and transfer to the outer shell of a medical filter. Leave a bit in the centrifuge tubes as this bit will contain the larger particles. Also, stay below the stepped edge of the medical filter (this is the maximum amount the filter can effectively filter). Then place the (closed) filter piece into the outer shell and press down until it clicks into the outer shell edge.	in bonding cleanroom	1 min	1	0.2 µl medical filter
10	Transfer the medical filter with solution to the bonding table and change gloves	in bonding cleanroom	1 min	1	Gloves
			4 min		

2.4 Clean the relevant side ("S3" or "S4") of the mass

Step	What	Where	Time	People	Tools

Step	What	Where	Time	People	Tools
11	Make sure the dry nitrogen supply is open and de-ionizing gun can blow a gentle consistent and well controlled flow. Make sure methanol and optical wipes are ready. Get the DI water running gently but solidly through the flexible hose Open petridish with bicarbonate of soda. Put that at the back to minimize risk of contaminating with cerium oxide Open petridish with cerium oxide. Put that in front of the bicarbonate of soda.	in bonding cleanroom	1 min	1	Dry nitrogen
12	Rinse DI water over side "S3"or "S4" of the TM. Rub the surface with a folded wet optical wipe embedded with cerium oxide paste in a random motion with moderate pressure. Make sure the actual bond area is rubbed best but extend the area to about three times the width and height. The cerium oxide paste should start sticking uniformly over the cleaned surface. The surface should become hydrophilic. Typically 30 seconds of rubbing is enough. Rinse again with DI water and rub with a clean optical wipe to get rid of most cerium oxide.	in bonding cleanroom	1 min	2	DI water, cerium oxide, optical wipes
13	Throw away the cerium oxide cloth and rinse fingers thoroughly.	in bonding cleanroom	1 min	2	
14	Rub side "S3"or "S4" with a folded wet optical wipe embedded with a copious amount of bicarbonate of soda paste with moderate pressure. Work from the inside out to work remainders of cerium oxide from inside out. Typically 30 seconds of rubbing is enough. Make the cleaning area slightly bigger than was done for the cerium oxide. Rinse with water to wash away bicarbonate of soda.	in bonding cleanroom	1 min	2	DI water, bicarbonate of soda, optical wipes
15	Repeat step 14.	in bonding cleanroom	1 min	2	DI water, bicarbonate of soda, optical wipes

Step	What	Where	Time	People	Tools
16	Rinse the cleaned area with methanol and blow dry gently with de-ionized nitrogen. Once again work from the clean inner area outward and cover a larger area. Wipe away as many water drops that have run along the barrel away with a cloth soaked in methanol. **Rinsing methanol over the surfaces coated with first contact HAS TO be avoided. The barrel and bonding flats should be wiped with acetone near coated surfaces for this reason. The bonding area should NOT be wiped with acetone. It HAS TO be wiped with methanol.**	in bonding cleanroom	1 min	2	Methanol, dry nitrogen, optical wipes
17	Inspect the cleaned surface: it is verified as cleaned when no particles or films are present when viewed without optical aid at a viewing distance of 5”- 6” while the surface is illuminated by a fiber optic light source against a dark background.	in bonding cleanroom	1 min	2	Fiber optic light source, magnifying glass (optional)
18	Change gloves	in bonding cleanroom	1 min	2	Gloves
19	Lift up the test mass using the ergo-arm and wipe carefully and thoroughly with a cloth soaked in methanol underneath the mass, where water ran.	in bonding cleanroom	5 min	2	Ergo-arm, methanol, optical wipes
20	Turn the mass 90 º, such that the cleaned side is pointing upward ready for bonding and cover with an optical wipe.	in bonding cleanroom	1 min	2	Optical wipes
21	Transport the mass on the ergo-arm to the bonding table and put it down onto the V-block for bonding.	in bonding cleanroom	5 min	2	
			19 min		

2.5 Clean ear

Step	What	Where	Time	People	Tools
22	Make sure step 11 is still true	in bonding cleanroom	1 min	1	First Contact, clean room wipes
23	Take the ear into an optical cloth and rinse with DI water. Embed another optical wipe with cerium oxide and rub the entire ear with moderate pressure. Make sure all edges and corners are covered. Take about 1 minute. Rinse again with DI water to get rid of most cerium oxide.	in bonding cleanroom	1 min	1	DI water, cerium oxide, optical wipes
24	Transfer the ear to another optical wipe embedded with cerium oxide and rub the bonding surface in particular. Do this for 30 seconds.	in bonding cleanroom	30 sec	1	DI water, cerium oxide, optical wipes
25	Transfer the ear to another clean optical wipe and rub to remove most cerium oxide. Make sure gloved hands are rinsed thoroughly as well.	in bonding cleanroom	1 min	1	
26	Transfer the ear to an optical wipe embedded with copious amounts of bicarbonate of soda and rub it with moderate pressure for about 1 minute. Rinse.	in bonding cleanroom	1 min	1	DI water, bicarbonate of soda, optical wipes
27	Transfer the ear to a fresh optical wipe embedded with copious amounts of bicarbonate of soda and rub in particular the bonding surface with moderate pressure for about 30 seconds.	in bonding cleanroom	30 sec	1	DI water, bicarbonate of soda, optical wipes
28	Thoroughly rinse the ear and gloved hands with DI water while the ear is lying in a hand upside down, ensuring to never touch the bonding surface with the gloves.	in bonding cleanroom	30 sec	1	
29	Rinse the ear with methanol and cover with an optical wipe.	in bonding cleanroom	30 sec	1	Methanol, optical wipe
30	Transfer part back to petridish on bonding table lined with a fresh optical wipe	in bonding cleanroom	30 sec	1	Petridish, optical wipe
31	Clean gloves	in bonding cleanroom	30 sec	1	Gloves

Step	What	Where	Time	People	Tools
			7 min		

2.6 Bonding

Step	What	Where	Time	People	Tools
32	Set the pipette to 9.6 µl and lock.		1 min		
33	Take the optical wipe off side 1 of the mass. Take a folded optical wipe soaked with methanol and wipe the bonding surface. Blow dry with dry nitrogen from the de-ionizing gun. Use the fibre optic light to thoroughly inspect the surface for small specks from a distance of 5-6”. Wipe and/or blow any specks away.	in bonding cleanroom	1 min		
34	Wipe down the bonding jig with methanol to remove any dust and place it on the bonding surface by referencing the front surface (“S1”) of the mass and lining the sliders up with the fiducials on surface (“S3”). Tighten the spring-loaded screw onto surface “S2”.	in bonding cleanroom	3 min	1	Optical wipe, methanol
35	Pick up the ear and wipe the bonding surface in one stroke with a folded optical wipe soaked with methanol. Gently blow dry with dry nitrogen using the de-ionizing gun. Use the fibre optic light to thoroughly inspect the surface for small specks from a distance of 5-6”. Wipe and/or blow any specks away. Lay the ear back down.	in bonding cleanroom	1 min	2	Dry nitrogen, de-ionizing gun, high intensity light source, optical wipe soaked in methanol
36	Put a pipette point onto the pipette and extract the required solution from the medical filter, making sure not to touch the pipette tip to anything except the solution and making sure to close the filter again. Give the surface of the mass one more glance to ensure no more specks have landed and then place the drop of bonding solution down about 3 cm from the left reference edge and 1 cm from the top reference edge of the bonding jig (middle of the ear). Remove the pipette tip and place the	in bonding cleanroom	1 min	2	Pipette, pipette tips bonding solution

Step	What	Where	Time	People	Tools
	pipette back.				
37	Pick up the ear. Give the surface of the mass one more glance to ensure no more specks have landed. Place the ear carefully onto the mass, while lining it up with the top-left corner of the bonding jig hole.	in bonding cleanroom	1 min	1	High intensity light source
38	Inspect how the bonding solution spreads. It should spread readily over the entire bonding surface. Viewing at a shearing angle of ~30 º one should see coloured fringes. There might also be bubbles visible. These should be moving towards the edges. Note down on the inspection sheet all relevant information including the state of the bond initially (bubbles and fringes). Keep on checking for about 5 to 10 minutes.	in bonding cleanroom	5-10 min	2	High intensity light source
			18 min		

2.7 Initial curing and inspection

Step	What	Where	Time	People	Tools
39	Inspect the bond every 30 minutes for the first 2 hours and note down on the inspection sheet.		25 min 5 min 25 min 5 min 25 min 5 min 25 min	0 1 0 1 0 1 0	Fibre optic light, pen, inspection sheet F1000006
40	Remove the bonding jig carefully after 2 hours		10 min	1	Fibre optic light, pen, inspection sheet F1000006
41	Inspect the bond every hour for the following 3 hours.		55 min 5 min 55 min	0 1 0	Fibre optic light, pen inspection sheet F1000006

Step	What	Where	Time	People	Tools
			5 min	1	
			55 min	0	
			5 min	1	
42	Leave bond to cure for at least 12 hours before starting the other side.		7 hrs		
			12 hrs		

2.8 Bond the other side

Step	What	Where	Time	People	Tools
43	Use the ergo-arm to move the mass back to the sink to allow for starting the bonding procedure for the other side of the mass	in bonding cleanroom	10 min	2	First Contact, clean room wipes
44	Repeat steps 1 through 38.	In office/ in bonding cleanroom	1 hr 46 min	1,2	See above
45	Repeat the curing steps 39 through 42	In bonding cleanroom	12 hrs	0,1	See above
46	Use the ergo-arm to store the mass back into it's cake-tin and move the cake-tin to mass storage	In bonding cleanroom/ mass storage place	30 min	2	Ergo-arm, cake-tin

2.9 Curing

Step	What	Where	Time	People	Tools
47	Leave the ears to cure in storage for at least 4 weeks before using them	Mass storage	28 days	0	

Step	What	Where	Time	People	Tools
	for the monolithic suspension				

3 Back-up procedures

3.1 To-do when the bond quality is too low within 8 hours after bonding – debonding

Defining too low quality:

If after bonding an ear it becomes obvious that:

1. (despite every caution) a particle is trapped in the bond, which is visible by the eye without magnifying aids, causing a clear dark speck with fringes around it.
2. an area of more than 50 mm^2 (area of ~ 7 x7 mm or ¼" x ¼") part of the bonding area is not covered by bonding solution. This can be seen by the fact that the area not bonded is reflective in nature, whereas the area bonded is transparent. The total bonding area is approximately 1185 mm^2, which means that the area not bonded can be maximally 4.2%. Bond loss measurements performed by Peter Murray (P0900053) were performed with a bond in which approximately the same percentage of bond area was not bonded.
3. If there is a number of small bubbles in the bond initially they can make up a slightly larger percentage of the bond area (up to 8% as it is likely that some or most of the bubbles will move to the sides and disappear. It is not necessary to debond immediately in that case.

It will take approximately 50 to 60 seconds for a bond to settle initially in a laboratory at 20 °C (humidity unknown but typically 30-50% in the laboratory) (Reid et al., Phys. Lett. A, 2007). Initial settling means that with minor force applied it will not move. In the first 8 hours after bonding it is possible to break to bond using a combination of rinsing with copious amounts of DI water and applying a moderate amount of manual force (preferably in a rotation around the axis perpendicular to the bond surface, effectively shearing it). For 1 inch bonded discs a 100% de-bonding success rate was achieved using this method up to 13 hours after the bond

was made (T1000464). As the ear bonds cover a larger surface area a shorter time period is recommended. The procedure for this is discussed below.

Step	What	Where	Time	People	Tools
1	Observe that the bond quality is unlikely to meet requirements.	In clean-room	10 min	2	High intensity light source
2	Carefully remove the bonding jig, by releasing the spring-loaded bolts and lifting it carefully off the surface.	In clean-room	1min	1	
3	Lift up the mass using the ergo-arm, transport to the washing table and rotate the mass 90° back before placing it down onto the V-block on the basin.	In clean-room	10 min	2	Ergo-arm
4	Run copious amounts of DI water along the bonded ear. Apply a moderate amount of manual force in a shearing direction or rotation. This combination should allow the ear to come off the side of the mass within 3 minutes.	In clean-room	1 min	2	DI water
5	Gently place the ear in a beaker with DI water.	In clean-room	1 min	1	Beaker with DI water
6	Keep on rinsing both the flat of the mass with copious amounts of water. Then clean the side of the mass again with cerium oxide and bicarbonate of soda as discussed in section 2.4 in the procedure.	In clean-room	19 min	2	DI water, cerium oxide, bicarbonate of soda, methanol, optical wipes, dry nitrogen, de-ionizing gun
7	Clean the ear again as discussed in section 2.5.	In clean-room	7 min	2	DI water, cerium oxide, bicarbonate of soda, methanol, optical wipes
8	Repeat the remainder of the bonding procedure discussed in sections 2.6 and 2.9.	In clean-room	1 hr 46 min 12 hrs	2 0	See above.

3.2 To-do when the bond quality is too low after a longer time or if debonding through rinsing doesn't work – ultrasonic debonding

In case no abnormalities were observed within the initial period where debonding by rinsing with copious amounts of water is still possible, there is still the possibility of debonding using ultrasonic stimulation in DI water up to (T1000464 and T1000465) 48 hours after the bond has been made. It is extremely unlikely the bond will change significantly for the worse after 12 hours.

Step	What	Where	Time	People	Tools
9	If the bonding jig has not removed yet, remove the bonding jig carefully, by releasing the spring-loaded bolts and lifting it carefully off the surface.	In clean-room	1 min	1	
10	Set-up the ultrasonic bath. Put blocks in the bottom to ensure the ear will be submerged completely, but the mass will be submerged by 1 cm at the most. Fill with DI water. Place the stiff lid onto the bath. Attach handles to the V-block and set-up the V-block on top of the ultrasonic bath lid.	In clean-room	15 min	2	Ultrasonic bath with lid, DI water, V-block plus handles
11	Lift up the mass from the bonding table using the ergo-arm, transport to the V-block on ultrasonic bath. Rotate the mass 180° so that the ear to be de-bonded is facing down and place it carefully on the V-block.	In clean-room	10 min	2	Ergo-arm, V-block, Ultrasonic bath
12	Carefully lift up the V-block with mass with 2 people while a third person removes the lid. Lower the V-block with mass down into the ultrasonic bath.	In clean-room	3 min	3	
13	Switch on the ultrasonic bath: temperature setting: 50° C, running the ultrasonic indefinitely until the ear falls off. If the procedure is commenced within 12 hours after bonding, it should take maximally 30 minutes for the ear to come off. Switch off the ultrasonic bath.	In clean-room	30 min	1	
14	If possible remove the ear from the bath and deposit in a beaker with DI water.	In clean-room	1 min	1	Beaker with DI water
15	Use the handles on the V-block to carefully lift the mass out of the bath with two people and a third person places the lid back onto the bath, before the two people put the mass back down onto it.	In clean-room	3 min	3	Lid

Step	What	Where	Time	People	Tools
16	Use the ergo arm to lift the mass onto the V-block on the washing table and rotate 90°. Also dry the barrel of the mass.	In clean-room	10 min	2	Ergo-arm
17	Clean the side of the mass again with cerium oxide and bicarbonate of soda as discussed in section 2.4 in the procedure above. It is likely that a more thorough clean with cerium oxide is necessary in particular along the edges of the old bond, because remnants of bonding solution are likely to be visible.	In clean-room	19 min	2	Optical wipes, cerium oxide, bicarbonate of soda, methanol, dry nitrogen
18	Select a new ear and repeat the bonding procedure from step 1 in section 2.1	In clean-room	1 min	1	

3.3 Applying/removing First Contact

3.3.1 Applying

Step	What	Where	Time	People	Tools
1	See E1000079.		3 hours	1	See E1000079.

3.3.2 Removing

Step	What	Where	Time	People	Tools
1	See E1000079.		3 hours	1	See E1000079.

3.4 Drag-wiping

Step	What	Where	Time	People	Tools
1	See E1000079.		3 hours	1	See E1000079.

4 Remarks

The old bonding procedure document E050228 uses a 1:4 ratio between sodium silicate solution and water in stead of 1:6. This has been changed by the findings of Elliffe et al and confirmed by ourselves in recent tests as the average strength of 1:6 bonds is somewhat higher and the standard deviation of the strength is lower.

The old procedure doesn’t use a centrifuge to get rid of large particles. We do.

The old procedure does use pipetting to take solution out of the bottle. We do not normally use this technique. We normally pour the solution into the 15 ml centrifuge tube.