LIGO Laboratory / LIGO Scientific Collaboration

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Silicate bonding procedure (Hydroxide-Catalysis Bonding)

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1 Introduction

1.1 Purpose and Scope

Hydroxide-catalysis bonding is the process by which a hydroxide, Na in our application, catalyzes the silica surface by hydration and dehydration.

This document describes the procedure to join two pieces of glass using this bonding technique referred to as “silicate bonding”.

Because the surfaces are required to be in close contact to bond, a flatness of λ/10 peak-to-valley is required on the surfaces to maximize bond strength.

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

Operator must be dressed in clean room attire.

Frock, boots, head cover, facemask, and approved cleanroom gloves are required.

1.2 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 Catalog 53511-588

Microcentrifuge tubes –Eppendorf – 1.5 ml (VWR catalog No. 20901-551)

VWR® MiniFuge Microcentrifuge - 120V, 50/60Hz - VWR catalog No. 93000-196

Microcentrifuge tubes storing rack – VWR Catalog No. 20901-675

Centrifuge tubes, polypropylene, graduated, 50 ml, VWR Cat. No. 21008-240

Centrifuge tube's rack, VWR cat. No. 21008-485

Medical Filter: Whatman Filter Uniprep 0.2UM PK50 UN113ENYL Filter

Eppendorf* epTIPS* Pipet Tips – Sterile PCR Clean Filter Tips 2-20 µl – 10 Racks of 96 Tips Cat. No. 47745-092

Alpha 10 wipes – VWR Cat. No. TWTX1010 – (case)

Gloves - VWR Certi-Clean Class 100 Latex Gloves or Accu Tech Ultra Clean 91300 Gloves.

Methanol - Reagent grade

Sodium bicarbonate

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

1.3 Version history

09/16/05: The first version of this document was written by Helena Armandula and reviewed by Caroline Cantley in 2005.

07/16/10: The document was updated due to advances in the hydroxide preparation procedure. Also, the recommended hydroxide ratio has changed. Strength measurements have shown that a 1:6 volumetric ratio of the commercially available sodium silicate solution to DI water gives stronger bonds on average than 1:4 solutions do (Elliffe et al. Class. Quant. Grav., 2005 and recent results Karen Haughian).

2 Surface Preparation

Hydrophilic surfaces with a high density of Si-OH groups are necessary for successful bonding. The parts to be bonded should be treated as follows:

2.1 Rinse the substrates under de-ionized (DI) water to wash off any particles that could scratch the surface.

2.2 Gently scrub the surface to be bonded with a folded wet tissue (Alpha 10) embedded with cerium oxide paste.

2.3 Rinse under DI water while scrubbing the surface with a clean tissue to ensure the complete removal of the cerium oxide.

2.4 Repeat steps 2.2 and 2.3

2.5 Next, gently scrub the surfaces to be bonded with a folded wet tissue (Alpha 10) embedded with sodium bicarbonate paste ($NaHCO_3$) – Sodium bicarbonate or baking soda is used to neutralize the cerium oxide.

2.6 Rinse, scrubbing the surface with a clean cloth for about 15 seconds under running DI water. Ensure that the $NaHCO_3$ is completely removed and the water sheets-off the surface.

2.7 Repeat steps 2.5 and 2.6

2.8 Rinse the surface with methanol

2.9 To dry, wipe the surface with a folded Alpha 10 tissue with methanol to remove any water to avoid drying marks.

2.7 The surface 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.

2.8 Keep the cleaned parts wrapped in a dry Alpha 10 tissue in a large cleaned Petri dish lined with HV foil until they are ready to be bonded.

2.9 Change gloves.

3 Prepare bonding solution

3.1 Pour 2 ml of the commercially bought sodium silicate solution into a 15 ml centrifuge tube. Close the sodium silicate bottle promptly to prevent contamination.

3.2 Top-up the centrifuge tube to 14 ml with DI water to create a 1:6 volumetric ratio of solution.

3.5 Close tube.

3.6 Shake well (for about 1 min) to mix.

3.7 Label and date the tube. The lifetime for the mixed bonding solution is a week when kept well capped.

3.8 Open the tube and poor the solution into 3 1.5 ml centrifuge tubes. Close all centrifuge tubes again.

3.9 Insert the small centrifuge tubes axi-symmetrically into the centrifuge and spin for approximately 30 seconds.

3.10 Take 2 of 3 tubes out of the centrifuge and poor the solution into 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.

3.11 Solution is ready to use.

3.12 Change gloves.

4 Bonding Procedure

The amount of solution used on this procedure is for bonding small surfaces ~0.5" dia.

The amount of solution needs to be adjusted to the size of the parts to be bonded. For advanced LIGO 0.8 µl/cm^2 is recommended. For a 0.5" dia surface this equates to 1.0 µl of solution.

NOTE: An excessive amount of solution may result in a weaker bond.

4.1 Wipe the bench's surface and ensure that the surrounding areas are free of particulate contamination.

4.2 Set the measuring dial of the pipette to 1.0 µl.

4.3 Take a folded optical wipe soaked with methanol and wipe the bonding surfaces. 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.

4.4 Insert a clean tip on the pipettor.

4.5 Open the medical filter. Without touching the sides of the small centrifuge tube, withdraw the solution from it and close it again.

4.6 Give the surfaces one more glance to ensure no more specks have landed and then place the drop of bonding solution down without touching the surface. Remove the pipette tip and place the pipette back onto the rack.

4.7 Bring the two surfaces to be bonded into contact (using an alignment jig if needed) and apply a small amount of pressure.

4.8 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 at a visible rate.

4.9 Very tiny bubbles do not affect the bond strength.

4.10 The parts will set in about 50 seconds.

4.11 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.

5 Bond inspection

5.1 Inspect the bond every 30 minutes for the first 2 hours and note down on the inspection sheet.

5.2 Inspect the bond every hour for the following 3 hours. It is unlikely the bond will change much after that.

5.3 Allow the bonds to cure for a few days before handling and allow them to cure for at least 4 weeks before loading them.

6 Debonding

If after bonding glass parts it becomes obvious that:

1. 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. >5% 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;
3. there is a number of small bubbles in the bond initially they can make up >10% of the bond area (this a larger percentage 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 be necessary to debond. If this observation is made within 8 hours after bonding, debonding can be achieved by running copious amounts of DI water along the bonded pieces. Apply a moderate amount of manual force in a shearing direction or rotation. This combination should allow the bond to come apart within 30 seconds.

Before making a new bond, the parts need to be scrubbed again; follow the steps from the surface preparation section above.