



SPECIFICATION

Silicate Bonding Procedure
(Hydroxide-Catalysis Bonding)

Table with 8 columns: APPROVALS, DATE, R EV, DCN NO., BY, CHECK, DCC, DATE. Rows include AUTHOR: Helena Armandula, CHECKED: C.Cantley, APPROVED: C. Cantley, DCC RELEASE.

1 Introduction

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 lambda/10 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.

1.1 Equipment and Materials

Filtered dry nitrogen

Ionizing gun

High intensity light source

DI water

Mini centrifuge VRW- capacity 6x 1.5 ml Cat. No. 20668-212

Pipettor with tip ejector - Variable volume, 100 - 1000 ml - Eppendorf - VWR Catalog 53511-582

Pipettor with tip ejector - Variable volume, 0.1 - 2.5 ml - Eppendorf 2000- VWR Catalog 53511-586



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Microcentrifuge tubes –Eppendorf – 1.5 ml VRW catalog No. 20901-551
Microcentrifuge tubes storing rack – VWR Catalog No. 20901-675
Centrifuge tubes, polypropylene, graduated, 50 ml, VRW Cat. No. 21008-240
Centrifuge tube's rack, VWR cat. No. 21008-485

Syringe-less filters – VWR Cat. No. 28297-108VWR

VWR Universal Tip Cat. No. 53510-082

Eppendorf – Ultra Micro tips Cat. No. 53511-598

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

Sodium Silicate Solution – from Aldrich Chemical Co., Inc.
(~14% NaOH, ~27% SiO₂ by weight)

Ultrasonic cleaner

2 Procedure

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:

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- 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 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.
- 2.5 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.
- 2.6 To dry, wipe the surface 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 in a large cleaned Petri dish lined with HV foil and covered until they are ready to be bonded.

3 Prepare bonding solution

- 3.1 Insert a tip on the large pipettor and draw 5 ml from the bottle of the commercially bought sodium silicate solution; exercise care not to touch the sides of the bottle.
- 3.2 Place the solution into the large (50 ml) centrifuge tube. Close the sodium silicate bottle promptly to prevent contamination.
- 3.3 Fill a clean glass container with DI water.
- 3.4 Use a large pipettor to transfer water to the sodium silicate solution to obtain 25 ml (1:4) of bonding solution.
- 3.5 Close tube.
- 3.6 Shake well to mix.
- 3.7 Label and date the tube.
Lifetime for the mixed bonding solution is a week when kept well capped.



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3.8 Using a fresh tip, extract ~1.0ml (or amount to be determined) of bonding solution from the tube and place it on a syringe-less filter.

3.9 Empty the solution from the filter into a small centrifuge tube. Close tube.

3.10 Centrifuge for 5 seconds.

Solution is ready to use.

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.

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

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

4.1 Thoroughly inspect the substrates to be bonded with a high intensity light source against a dark background. If particles are seen, wipe the part with a folded Alpha 10 tissue embedded with methanol. Place substrates on top of a clean Alpha 10 wipe.

4.2 Insert a clean tip on the small pipettor and set the measuring dial to ~2 μ l,

4.3 Without touching the sides of the small centrifuge tube, withdraw the solution.

4.4 Dispense the bonding solution from the pipettor onto the glass without touching the surface.

4.5 Bring the two surfaces to be bonded into contact and apply a small amount of pressure.

4.6 Wiggle them slightly back and forth if bubbles are present to push air bubbles away.

4.7 Very tiny bubbles do not affect the bond strength.

4.8 Align the parts to each other. A bonding wave will be observed; within a few minutes the wave will be spread out through the entire area.

4.9 The parts can be moved only for a few seconds to be aligned until they set.

4.10 If a particle or large bubble is observed and there is a need to pull the substrates apart, immerse in a beaker with DI water with a small amount (10% solution) of detergent (Micro90) added, then place this beaker into an ultrasonic bath. Care should be taken to not leave in the detergent or ultrasonic for long periods as this can degrade the surface roughness of the samples.

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

4.12 Dispose of leftover solution after bonding by generously flushing the tube with water.

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