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Shear strength testing of hydroxide catalysis bonded discs

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Abstract

Discs with a diameter of 25 mm were bonded in September 2010 by three different people during a training excercise in Hanford using hydroxide catalysis bonding.

All 26 discs were cured according to the standard 4 week room temperature procedure. 6 of the discs then were exposed to the aLIGO UHV clean bake procedure (125 degrees C for 48 hrs in Ultra high vacuum). All discs were sent back to Glasgow, where a large number of them was strength tested in shear.

It was found that the strength of the discs (without the clean bake) varied somewhat between the three people. 11, 13 and 16 MPa was found, with a standard deviation of 5 MPa for the two lower values and 3 MPa. The bonds that were UHV clean baked were loaded up to 25 MPa and none broke.

One of the six UHV baked samples was not strength testing but sliced into four slices. These slices were polished to conduct bond thickness measurements on it. This particular sample had some bubbles in it, which has presumably resulted in a wedged bond ranging from 35 to >380 nm bond thickness.

1 Introduction

1.1 Purpose and Scope

Hydroxide catalysis bonding is used in aLIGO for bonding the ears (interface pieces) to the sides of the test masses and penultimate masses in the quadruple input and end suspensions. The interface pieces and bonding procedure have been designed and developed at the Institute for Gravitational Research in Glasgow and applied in the prototype quadruple suspension for aLIGO. The know-how of this hydroxide catalysis bonding procedure however, needs to be transferred to the technical personnel at the aLIGO sites in Hanford (Washington state) and Livingston (Louisiana) for the purpose of applying this technique on 16 suspensions (32 masses, 64 bonds).

For this purpose a training exercise was held from 13th to 23rd September 2010 in Hanford to train three aLIGO people (Gerardo Moreno, Betsy Bland and Margot Phelps) on how to use the technique of hydroxide catalysis bonding. This involved bonding a large number of silica discs (∅25 mm x 3 mm) for each of them to master the minute details of the procedure. Each of them made 15 bonds or more.

To verify the strength of the bonds they made, the discs were sent to Glasgow to be strength tested in shear on the 2nd February 2011. A small selection (2 bonded discs per person) was vacuum baked (according to the vacuum cleaning procedure for aLIGO (E960022) in Hanford prior to being sent to Glasgow. This was done to see if the bonds are affected (in quality and strength) by this cleaning procedure. As it stands this cleaning procedure is not required on the silica parts of the suspensions for other reasons. However, it would be useful to know for possible future requirements.

This report summarises the observations made during the bonds made by the trainees and is an account of the shear strength testing performed after cure (with and without vacuum heat treatment).

1.2 References

E960022	LIGO Vacuum Compatibility, Cleaning Methods and Qualification Procedures
E050228	Silicate bonding procedure (Hydroxide-Catalysis Bonding)
Physics Letters A 374 (2010) 3993–3998	Re-evaluation of the mechanical loss factor of hydroxide-catalysis bonds and its significance for the next generation of gravitational wave detectors, L Cunningham et al.

1.3 Version history

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2 Procedure

2.1 Bonding samples

The bonds were made by bonding together fused silica discs (∅25 mm x 3 mm) supplied by Edmund optics (with a flatness stated to be λ/10 at 633 nm). The flatness of the individual discs was not measured before bonding, so any effect of the flatness on either strength or bond thickness cannot be determined. The procedure for hydroxide catalysis bonding described in E050228 was followed.

The three main steps in this procedure are:

1. Cleaning the samples: two times with a rub with a cerium oxide and water paste, two times with a sodium bicarbonate and water paste, followed by a rinse with spectroscopic grade methanol.
2. Preparing the bonding solution: one part of Sigma-Aldrich sodium silicate solution (~10.6% NaOH and 26.5% SiO_2) and six parts of (18 MOhm) DI water. This is mixed through manually shaking the solution, then centrifuged at 10 000 RPM to make the larger particles separate to the bottom, followed by a filtration with a 0.2 μm sterile Whatman medical filter, which ensures the solution ends up in a sterile and sealed volume free of any other contaminants.
3. Bonding: The pipette was set to 4 μl (0.8 μl/cm^2 for 5 cm^2). The samples were wiped with methanol one more time and blown dry with ultrapure dry nitrogen and inspected for specks prior to applying the bonding solution with a pipette to one part and placing the other part on top to create the bond.

The quality of the bonds was monitored in the first few minutes after making the bond on special sheets and the bond quality was also noted down several months after the bond was made just prior to strength testing. The samples were left out on the flow bench table in the laboratory at ~21°C for between one and five days and were then packed in foam and cotton bags and left in the same laboratory to cure.

2.2 Vacuum bake treatment

Six of the bonded samples were exposed to a vacuum clean bake treatment in December 2010 in which they were put in a vacuum oven for 48 hours at 120°C according to the clean bake procedure described in E960022. The pumps that create the vacuum during the vacuum bake are rated to 10^{-9} mbar.

2.3 Strength testing

After the samples were vacuum baked or just left at room temperature for several months, they were all shipped to Glasgow (the samples cured at room temperature only were shipped with DHL, the vacuum baked samples were brought across by Travis Sadecki in his suitcase) and then strength tested in early February 2011 using a shear strength set-up. In this set-up each bonded disc is clamped into two steel sample holders. Because the discs in these tests are only 3 mm thick, but the set-up is designed for discs with a thickness of 6.3 mm, aluminium discs were glued to the back and front of each of the

bonded discs with double-sided tape. The circumference of the discs was also taped with a soft black electrical tape to allow for clamping with an even load around the circumference of the sample from the clamping (if this is not done the risk of creating high local stress concentrations due to hard to hard material contact of rough surfaces would be too high).

The holder with sample were slid together into the shear strength set-up which is installed on a Zwick 250 kN strength testing machine (Figure 2.1).

Figure 2.1 Shear strength testing set-up

The sample was loaded in shear by moving the loading bar away with a rate of 1 mm/min. The load was recorded with a 50 kN load cell. The measurement was stopped when the load drops to 80% the maximum load achieved in the test or if the load became higher than approximately 12 kN for safety reasons and because this bends the set-up itself so much that the loading conditions on the sample change.

Nineteen of the samples cured at room temperature only and five of the samples that were vacuum baked were strength tested.

2.4 Bond thickness measurements

The sixth vacuum baked sample (sample B9) was sliced into four slices of a thickness of approximately 2 mm using a 0.25 mm thick steel disc saw with diamond embedded in it. The four slices were then polished using a Logitech polishing machine working from a 600 grit silicon carbide grit, to a 9 µm aluminium oxide powder to a 3 µm aluminium oxide powder and finishing off with a chemical polish with Syton, resulting in polished surfaces with a RMS roughness of approximately R_a = 5 nm.

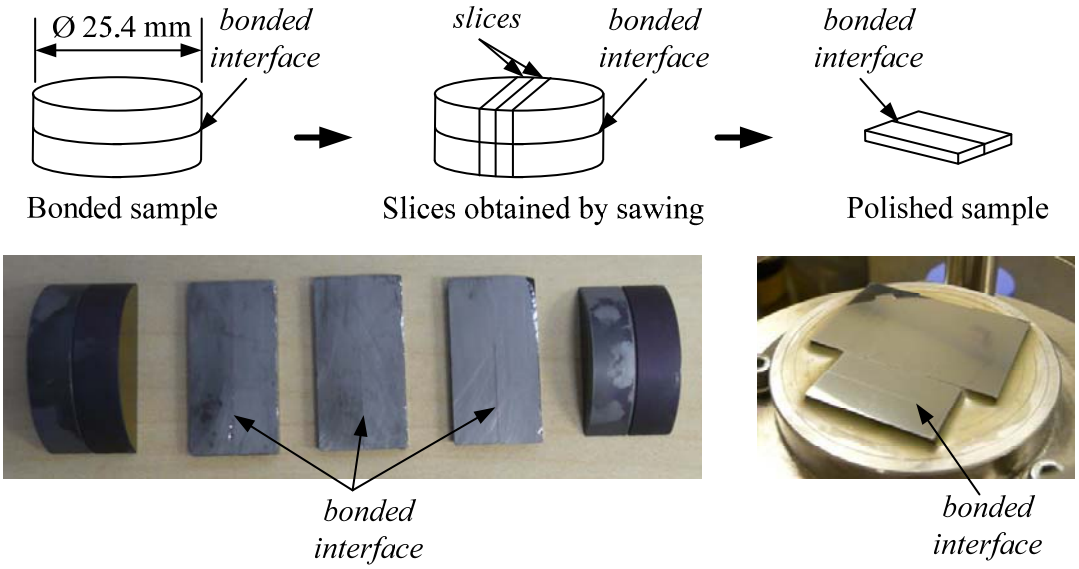


Figure 2.2 Example of preparation procedure for SEM imaging a bonded set of discs (in this image the sample is a silicon-silicon bond)

The samples were then coated with very thin layer of gold (~ 10 nm) to improve electrical conductivity of the samples to prevent/minimize charging effects during the SEM imaging process. The samples we scored across the bond in a large number of locations along the entire length of the samples as shown in to allow for a close-up look at the bond and measure its width using the FEI Nova 200 Dualbeam Scanning Electron Microscope at the Kelvin Nanocharacterisation Centre at the University of Glasgow. The added benefit is that it is easier to locate exactly where in the sample the measurement was taken.

A B C D

Figure 2.3 Polished slices of sample B9 with scores in the gold pattern (the photograph was taken after imaging, which is why large patches of the gold coating are missing. During SEM imaging a copper sticky tape was connected to the samples in those locations in order to ground them.)

3 Results

3.1 Bonding

The quality of the bonds was assessed by looking at the location and size of bubbles in the bonds after the long period of curing (and after vacuum bake). Drawings were made of all the bonds to indicate features and are shown in Figure 3.1.

A few samples not only had bubbles, but also had specks: B8, B12 and MP9. The speck in MP9 was visible before bonding and left there on purpose, to show the trainees the effect of a speck on bond quality. Some discs are marked with a (V), indicating that they were vacuum-baked.

3.2 Vacuum baking

Samples MP11, MP12, B9, B2, G10 and G11 were vacuum baked using the procedure described in section 2.2

The bond quality of all samples was noted before and after the vacuum baking had been conducted. There was no notable change between before and after bonding.

3.3 Strength testing

An overview of the strength testing results is given in Figure 3.2, in which the maximum load measured in N has been converted to a maximum shear stress by dividing the force by the bond surface area. More detailed results can be found in appendix A, in which all data can be found in tabulated format.

By looking at Figure 3.2 a number of observations can be made.

Figure 3.1 Bond quality of all bonds made

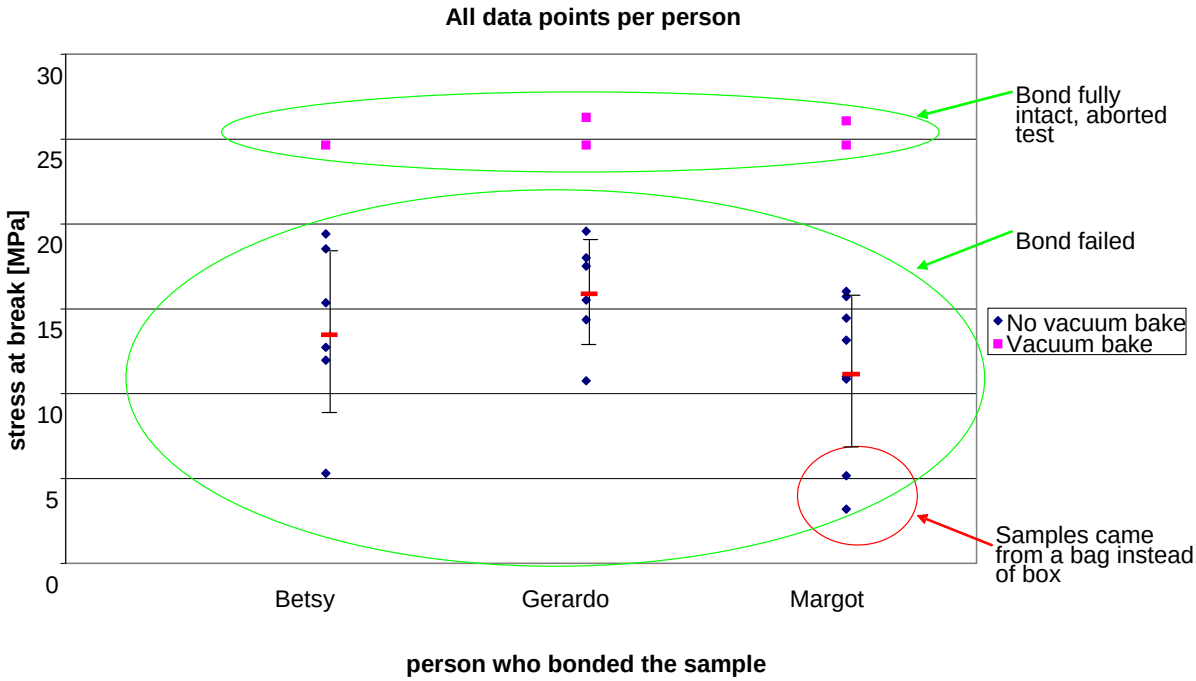


Figure 3.2 Strength testing results for all samples with stress at break as a function of the person who bonded the sample.

Firstly it can be noted that all samples that were vacuum baked show a significantly higher strength than any of the samples that were not vacuum baked. In fact these samples never broke across the bond. In all five vacuum bake cases the test was aborted, because at 12000 N, which is equivalent to ~25 MPa shear stress in the bond, the set-up itself was in fact bending so much that it wasn't deemed safe to continue the tests. On inspection afterwards, the edges were damaged because of the high contact load there, but the bulk of the samples including the bond appeared to be undamaged.

For all other samples the test was continued till brake and in fact all apart from the three samples with very low strength, disintegrated completely into an uncountable number of pieces. Upon inspection the bonds in these samples were broken as well as the bulk material. The three samples with strengths below 10 MPa were primarily broken across the bond but tended to have a number of factures in the bulk as well. An example of this can be seen in Figure 3.3. No significant differences have been observed between these three low strength samples and the other samples. All three were bonded over 98.5% of their surface area and no observations were made during bonding that could explain the low strength. The only difference between the two low strength samples bonded by Margot was that they were stored in a plastic bag just prior to strength testing, whereas all other samples were stored in a plastic box at this point. It seems unlikely that this is the cause of those low strength breakages though.

All non-vacuum-baked samples broke at equivalent stresses averaging 13.4 MPa with a standard deviation of 4.7 MPa. The large standard deviation is caused by three very low strength samples. The reason for the low strength of these samples is unknown. Bond

quality certainly is not a factor in this. If the three samples are left out of the average calculation the average strength found is 15.0 MPa ± 3.0 MPa.

The average strengths and standard deviation on the measurements of each of the three people are 13.9 ± 5.2 MPa for Betsy, 16.0 ± 3.1 MPa for Gerardo and 11.2 ± 4.8 MPa for Margot. The average strength found for Gerardo's bonds is somewhat higher than for Betsy and Margot, but their averages are pulled down by the three low strength samples and consequently their standard deviations are higher as well.

No correlation was observed between strength and percentages bonded; the two samples that were strength tested and bonded over less than 95% of their surface area, failed at between 10 and 15 MPa.

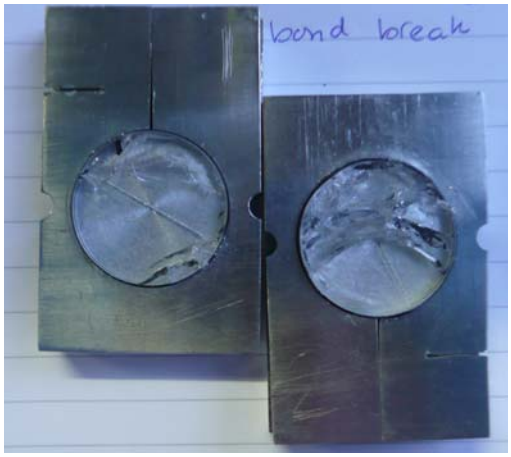


Figure 3.3 Disc MP2 broken at ~ 5 MPa

3.4 Bond thickness measurements

As explained in section 2.4 the four gold coated slices with lines scored in them were image with an SEM. From about half the number of locations it was possible to get a clear image of the bond when using the SEM in non-immersion mode with the beam energy at 10 keV and beam current at 0.13nA.

A typical SEM image obtained at two magnifications is shown in Figure 3.4.

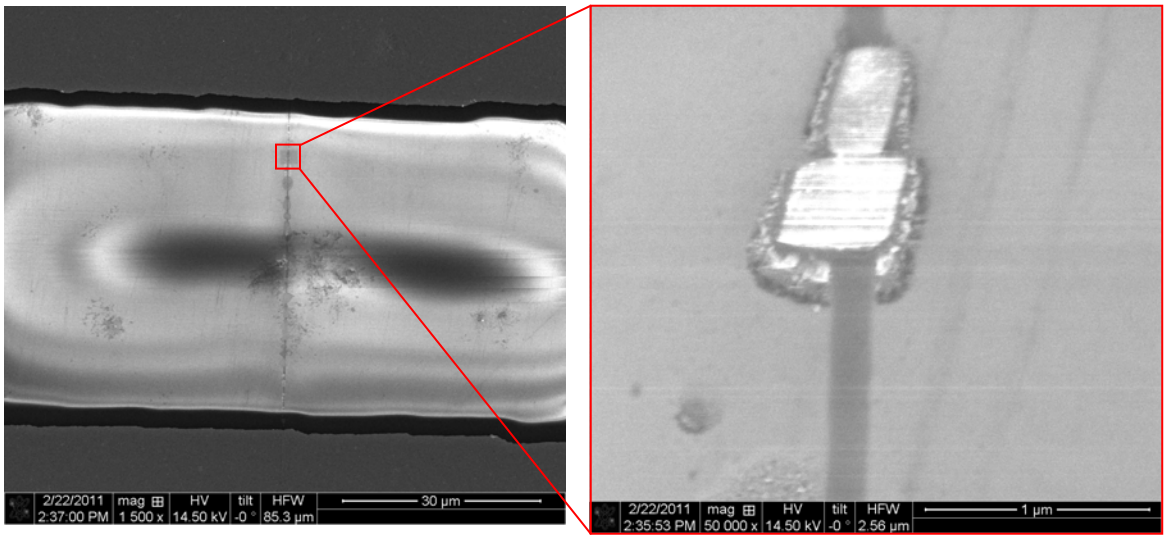


Figure 3.4 Typical SEM images (slice A, middle 4) of the bond at 1500x and 50,000x magnification

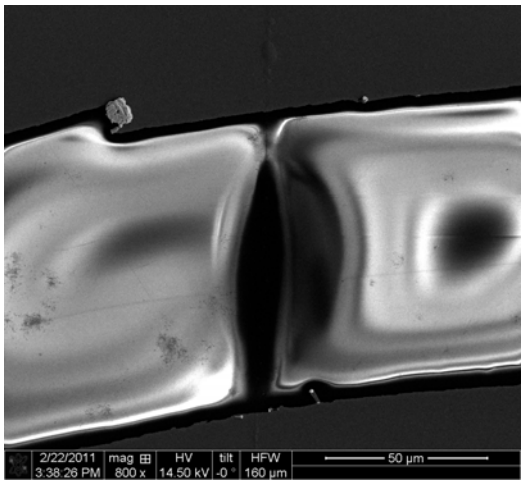
The right hand image was used in the SEM software to make a measurement of the width of the bond (in this case 150 nm). The measurement accuracy in these individual measurements was ± 10 nm.

The left hand image shows the full width of the score with a clear vertical line through which is the bond. The top and bottom dark grey areas are the gold coating on top of the silica.

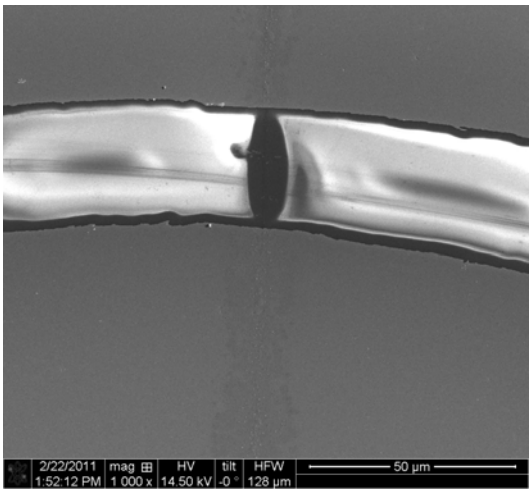
The images also show other features typically seen in all images. These are circular features sitting on top of the bond. These obscure the bond. In the right hand image a white feature which is more square in nature is sitting over the bond. It is thought that these features are remnants of the polishing compounds. As the bond is thought to be softer than the surrounding silica material, it is postulated that the polishing compounds have a tendency to accumulate around the bond and it is not easy to clean all of it away. The features are very helpful in achieving a good image of the bond itself (in aiding with focus and eliminating astigmatism).

There are also some speckled features sitting around the bond and elsewhere on the sample. These features are thought to be surfaces that are not quite smooth.

Finally, the larger oblong bands of changing brightness in the exposed silica area in the left hand image are thought to be charging effects on the image. An interesting point to observe here is that for thin enough bonds (like in this case) these charging features continue across the bond with some minor change in shape. The thicker the bond became as is the case in Figure 3.5 a) the more the charging effects became separated into two patches on either side of the bond. In Figure 3.5 b) it had in fact become impossible to get a good contrast image of the bond in higher magnification presumably because of this effect.



a) Bond thickness measured : 260 nm (slice C, middle 3)



b) Unable to measure bond thickness (slice C, bottom 2)

Figure 3.5 Images of slice C in two different locations to show the difference in charging behaviour.

Figure 3.6 shows an overview of the approximate (± 1 mm) positions of the thickness measurements made with respect to the full size original disc and where possible a number for the thickness found.

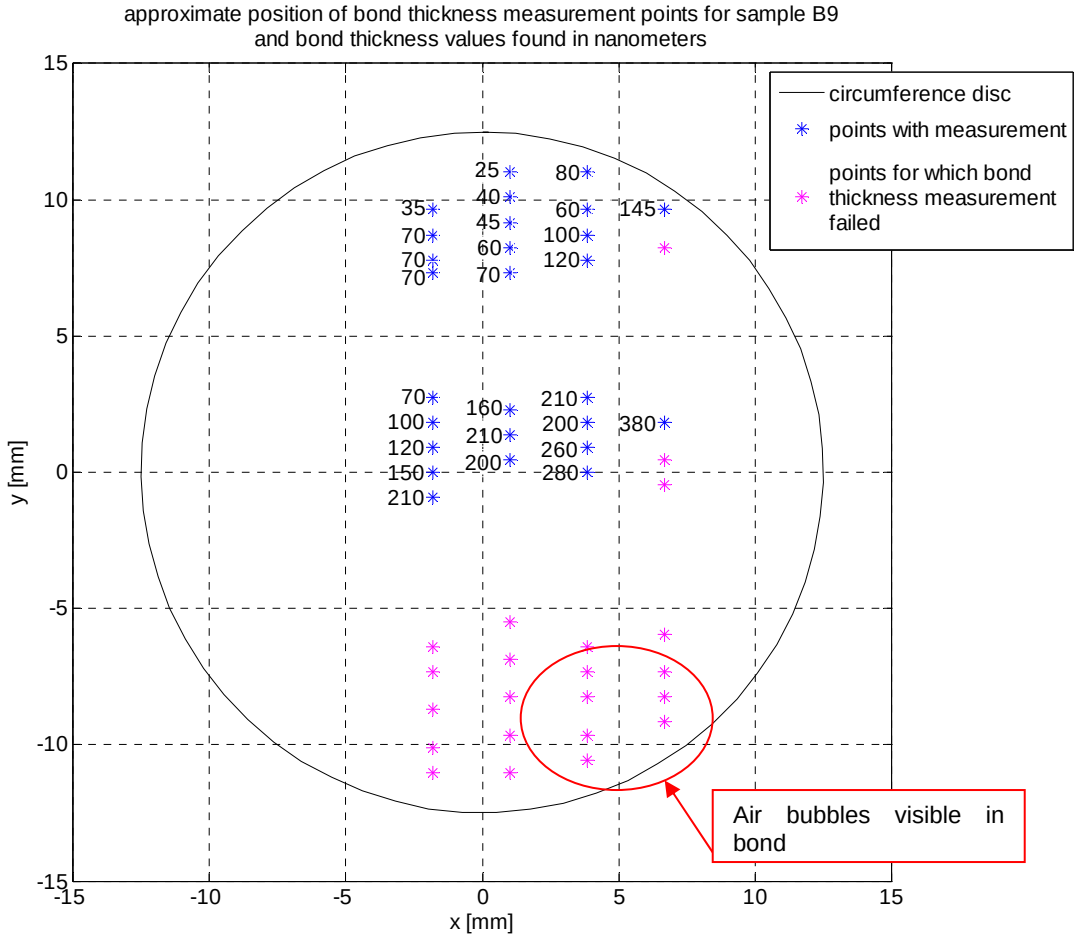


Figure 3.6 Approximate position of bond thickness measurement points for sample B9 and bond thickness values found in nanometers.

At the top of the sample the measurements show that the bond is very thin; around 25 nm, but moving down and somewhat to the right the bond becomes very wide until in the bottom it was not possible to make any thickness measurements presumably because of charging effects due to an even higher bond thickness (this wedge shaped bond is visualized in Figure 3.7 as well). If one looks at Figure 3.1 and looks up sample B9, one will find that this sample did have some bubbles in. The approximate location of the bubbles is indicated in Figure 3.6 as well. It appears that the bubbles might have had some effect on the bond thickness here.

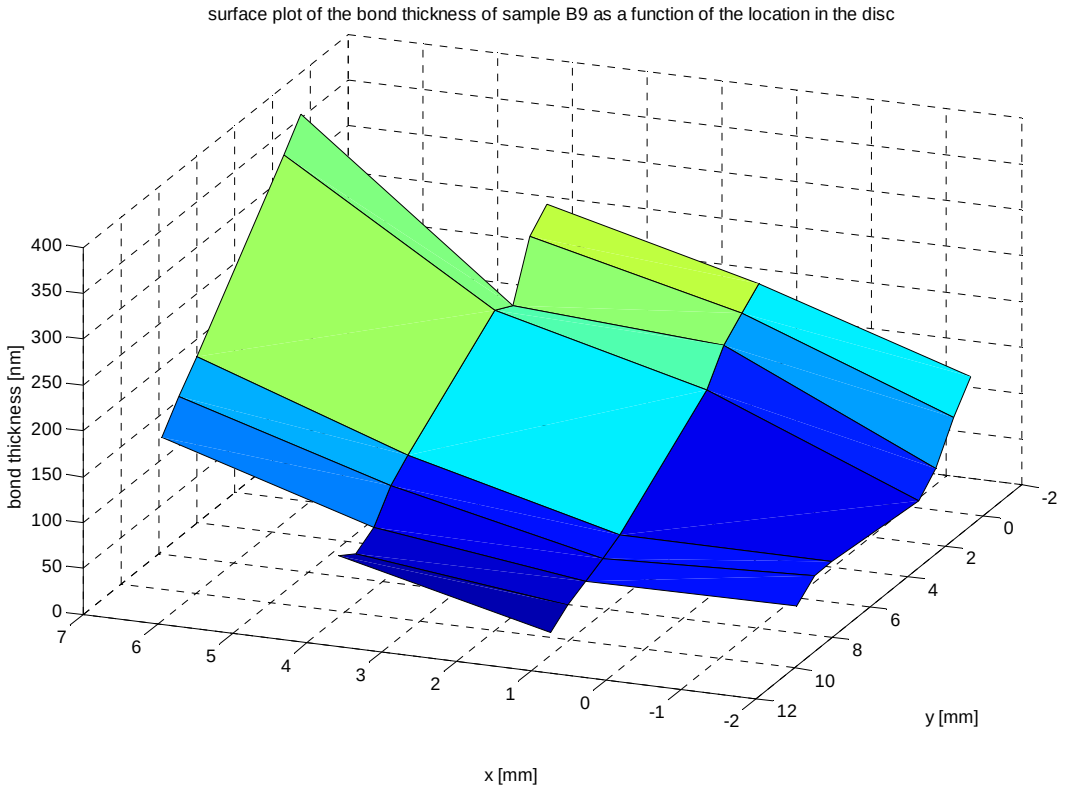


Figure 3.7 Surface plot of interpolated bond thickness values at set intervals. In this image the measured bond thicknesses have been interpolated to set locations (close to where measurements have been taken) using the datagrid function in Matlab and plotted as a surface plot to illustrate that the bond appears to be wedged.

4 Discussion and conclusions

Of 35 discs bonded in the week of the 14th September 2010, all were cured at room temperature for several months. Six of the bonded discs were subsequently vacuum-baked in the aLIGO vacuum baking facilities in Hanford at 120ºC for 48 hours. The vacuum baking did not have any influence on the visual appearance of the bonds.

Five of the vacuum baked samples and 20 of the remaining samples were shear strength tested using a Zwick 250 kN strength testing machine at the University of Glasgow by applying a shearing load on the bonds.

The tests of all five vacuum baked samples were aborted at an equivalent shear stress of 25 MPa for safety reasons without breaking the bonds. All other samples broke at much lower equivalent stresses averaging 13.4 MPa with a standard deviation of 4.7 MPa. The large standard deviation is caused by three very low strength samples. The reason for the low strength of these samples is unknown. Bond quality certainly is not a factor in this. If the three samples are left out of the average calculation the average strength found is 15.0 MPa ± 3.0 MPa. The average strength found is very good and comparable to 16 MPa average strengths found in 4-point bending tests of fused silica bonded using the same

technique. Gerardo has produced the strongest bonds on average, followed by Betsy and Margot. Once again no correlation could be found with bond quality because Margot produced significantly more clear bonds than Betsy or Gerardo did.

It is worth it to point out that no samples broke with an equivalent stress under 5 MPa, where the aLIGO ears will be loaded with an equivalent shear stress of 0.17 MPa.

For the first time a map of the bond thickness in a sample was made over a large portion of the bond area using a FEI NOVA scanning electron microscope. This sample was vacuum baked and the aim was to see if this sample had a different bond thickness than the bond thicknesses found previously and reported in Cunningham et al. (Physics Letters A 374 (2010) 3993–3998) of 40 to 100 nm. It was found that this sample had a wedged bond ranging from 25 nm to 380 nm (and possibly more). The wedge was probably caused by air bubbles trapped inside the bonded. It can therefore not be used as evidence for any influence of the vacuum baking on bond thickness, though it appears unlikely that the vacuum baking influenced the thickness of this wedged bond.

Appendix A

Table A.1 Data table of all strength testing results

Dics no.	Test no.	Bonded by	Max force	% bonded	Offset	Bond break/abort	Surface area	Stress at max force	Vacuum bake	Remarks
			[N]				[mm^2]	[MPa]		
MP8	1	Margot				abort	490.87	0		Set-up was not stiff enough, samples sliding out.
MP8	2	Margot	6460	0.75		break	490.87	13.16	no	Sample completely desintegrated
MP9	3	Margot	5330	0.6		break	490.87	10.86	no	Sample completely desintegrated
B6	4	Betsy	9530	1		break	490.87	19.41	no	Sample completely desintegrated
G1	5	Gerardo	8840	1		break	490.87	18.01	no	Sample completely desintegrated
G11	6	Gerardo	12900	0.99		abort	490.87	26.28	yes	Bond intact, edges broken
MP10	7	Margot	7720	0.995		break	490.87	15.73	no	Sample completely desintegrated
B10	8	Betsy	2610	0.985	yes	break	490.87	5.32	no	Bond break, sample desintegrated but not into millions of pieces
G6	9	Gerardo	9610	1	yes	break	490.87	19.58	no	Sample completely desintegrated
G10	10	Gerardo	12100	0.995		abort	490.87	24.65	yes	Bond intact, edges broken
G9	11	Gerardo	8600	0.99		break	490.87	17.52	no	Sample completely desintegrated
MP11	12	Margot	12100	0.995		abort	490.87	24.65	yes	Bond intact, edges broken
MP12	13	Margot	12800	1		abort	490.87	26.08	yes	Bond intact, edges broken
B2	14	Betsy	12100	1		abort	490.87	24.65	yes	Bond intact, edges broken
G2	15	Gerardo	5280	0.995		break	490.87	10.76	no	Sample completely desintegrated
G8	16	Gerardo	7050	0.98		break	490.87	14.36	no	Sample completely desintegrated
G4	17	Gerardo	7620	0.97		break	490.87	15.52	no	Sample completely desintegrated
B5	18	Betsy	7540	0.99		break	490.87	15.36	no	Sample completely desintegrated
B7	19	Betsy	9100	0.995		break	490.87	18.54	no	Sample completely desintegrated
B3	20	Betsy	6250	1	yes	break	490.87	12.73	no	Sample completely desintegrated
B11	21	Betsy	5880	0.985		break	490.87	11.98	no	Sample completely desintegrated
MP7	22	Margot	7100	1	yes	break	490.87	14.46	no	Sample completely desintegrated
MP5	23	Margot	5410	0.995		break	490.87	11.02	no	Sample completely desintegrated
MP4	24	Margot	7870	1	yes	break	490.87	16.03	no	Sample completely desintegrated
MP2	25	Margot	2540	1		break	490.87	5.17	no	Bond break, discs largely intact, taken from the bag instead of the box

MP3	26	Margot	1570	1		break	490.87	3.20	no	Bond break, discs largely intact, taken from the bag instead of the box