LASER INTERFEROMETER GRAVITATIONAL WAVE OBSERVATORY

LIGO Laboratory / LIGO Scientific Collaboration

LIGO-T1000464-v1 *ADVANCED LIGO* 4th August 2010

Preparation of an end or input penultimate mass (ETM/ITM PM)

(Hydroxide-Catalysis Bonding of ears and gluing prisms and magnet flags)

Rebecca Douglas, Mariëlle van Veggel

Distribution of this document:
DCC

This is an internal working note
of the LIGO Project.

California Institute of Technology
LIGO Project – MS 18-34
1200 E. California Blvd.
Pasadena, CA 91
Phone (626) 395-2
Fax (626) 304-98
E-mail: info@ligo.calt

Massachusetts Institute of Technology
LIGO Project – NW22-295
lbany St
e, MA 02139
7) 253-4824
) 253-7014
@ligo.mit.edu

Institute for Gravitational Research
University of Glasgow
Kelvin Building
Glasgow G12 8QQ
Phone: +44 (0)141 330 3340
Fax: +44 (0)141 330 6833
Web: www.physics.gla.ac.uk/igr/

LIGO Hanford Obse
P.O. Box 1970
Mail Stop S9-0
Richland WA 99352
Phone 509-372-8106
Fax 509-372-8137

ton Observatory
Box 940
n, LA 70754
Phone 225-686-3100
Fax 225-686-7189

http://www.ligo.caltech.edu/

De-Bonding Experiments on Samples Cured for up to Forty-Eight Hours

-Rebecca Douglas-

This experiment involved the bonding of silica disc samples using hydroxide catalysis bonding and de-bonding them after up to forty-eight hours of curing. Data was collected at several intervals over the forty-eight hour period. The discs were de-bonded using one of two methods; firstly by rinsing in DI water and secondly by using a variety of cleaning solutions in an ultrasonic bath. It was found that in general a longer curing time leads to a longer de-bonding time, but also that a more perfect bond (i.e. one that contained fewer bubbles) would require a longer time to de-bond.

1. Introduction

1.1 Gravitational Waves

Gravitational waves are predicted by Einstein's theory of general relativity[1]; they are waves in the fabric of space-time which propagate through the Universe at the speed of light[2]. As these waves travel, they distort the shape of the objects they pass through[3], in theory these distortions could be detected using a sufficiently sensitive interferometer.

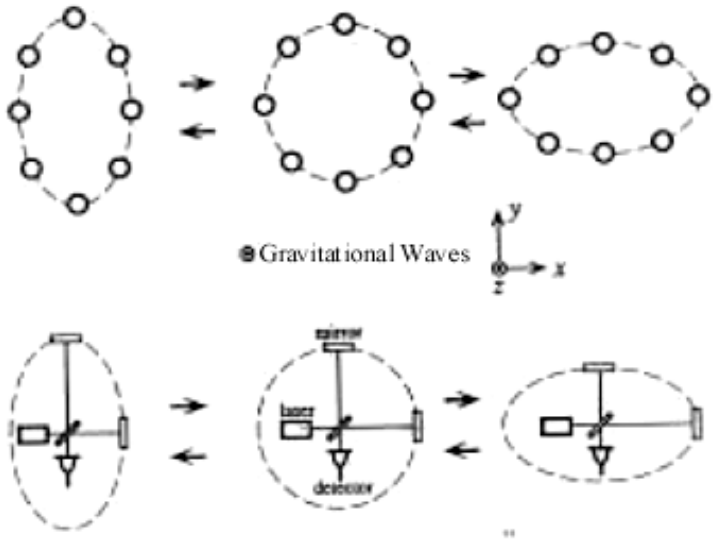

Fig1.1. As a gravitational wave passes across the object it causes a distortion – first in one direction, then in the next – changing the shape of the object. In the same way, as it passes across the interferometer, it changes the arm-lengths. This effect could be measured by the interferometer.

The main difficulty lies in building an interferometer that is sensitive enough to detect the distortions caused by gravitational waves. For an interferometer with an arm length of 1 km, the sensitivity must be good enough to detect a distance change on the order of 10^{-18} m[4]. Ground-based detectors such as LIGO and GEO600 attempt to tackle this challenge, as will Advanced LIGO. LISA is on a much larger scale, and will aim to perform the same task in space[5] – free from disturbances such as tectonic activity.

1.2 Advanced LIGO

Advanced LIGO is a large gravitational wave detector expected to replace LIGO (Laser Interferometer Gravitational-Wave Observatory) in 2014[5]. Its basic form is that of a very precise interferometer.

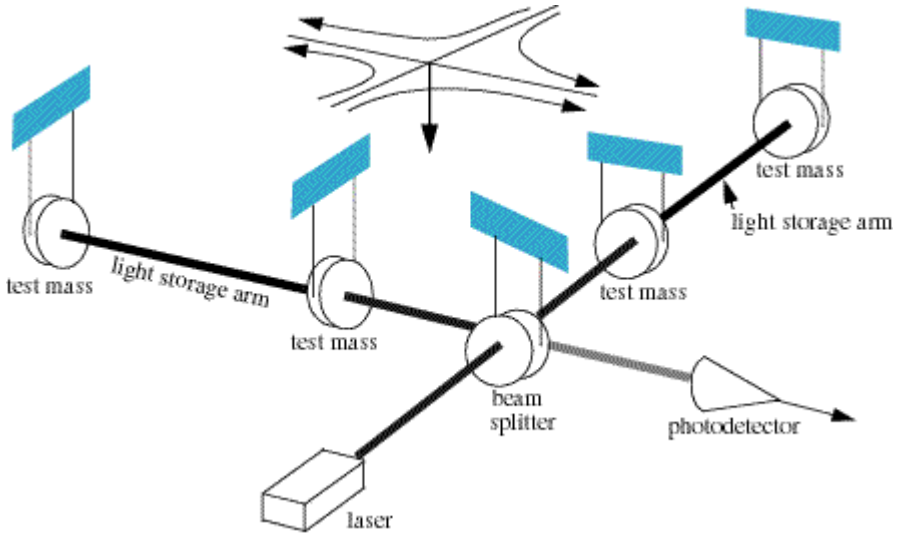

Fig1.1. Diagram of the Advanced LIGO interferometer.

The test masses and the fibres supporting them will both be made from silica. The fibres are attached to the test masses by silica "ears." One possible method of attaching these ears to the test masses is hydroxide catalysis bonding. The advantage of this being that the bond which forms is of a very similar material to the silica it holds together.

This kind of bonding takes four weeks to reach its maximum strength[7]. It is, however, already very strong well before that time.

During assembly of these aLIGO suspension there is a small chance that something might going wrong with either the bond or the ear. The mostly likely events to happen are:

1) that the bond is not good enough (part of the bond is not forming) or the ear is not aligned. This can be discovered in the first couple of hours after bonding.
2) that the ear gets so badly damaged during other installation procedures that it cannot be used in the suspension anymore.

In both these cases the ear will need to be de-bonded.

Experiments done by Karen Haughian (T1000xxx) and Marielle van Veggel have shown it is possible to de-bond samples with bonds up to 48 hours ultrasonically in a 10% solution of Micro-90 in water, but that it become difficult after that. Also some pre-liminary studies have shown very fresh bonds can be de-bonded under flowing DI water, which is logistically are more interesting solution for debonding in the first case stated above.

The aim of this experiment is to study de-bonding after the initial bond has been formed by rinsing under DI water and ultrasonically. If a bond is badly formed or flawed – or if damage occurs later – it will be useful to know if it can be removed, as well as how this should be done and what the effect on the silica will be.

For the rinsing under DI water we would primarily like to learn how long after bonding the sample it is still possible to reliably debond.

For the ultrasonic tests we would primarily like to learn if different detergents or chemicals are more effective than other at debonding for bonds that are up to two days old.

2. Method

2.1 Samples

The samples used in these experiments were Corning 7980 fused silica discs with diameter 25 mm and thickness 5 mm.

2.2. Flatness testing

Initially the flatness of the discs was tested, since flatness effects bond quality. These tests were performed using a Zygo interferometer, and no discs with a flatness value of greater than 95 nm were used in the experiment.

These values of flatness before bonding were later compared to those after de-bonding. This allowed any damage caused by the process to be highlighted.

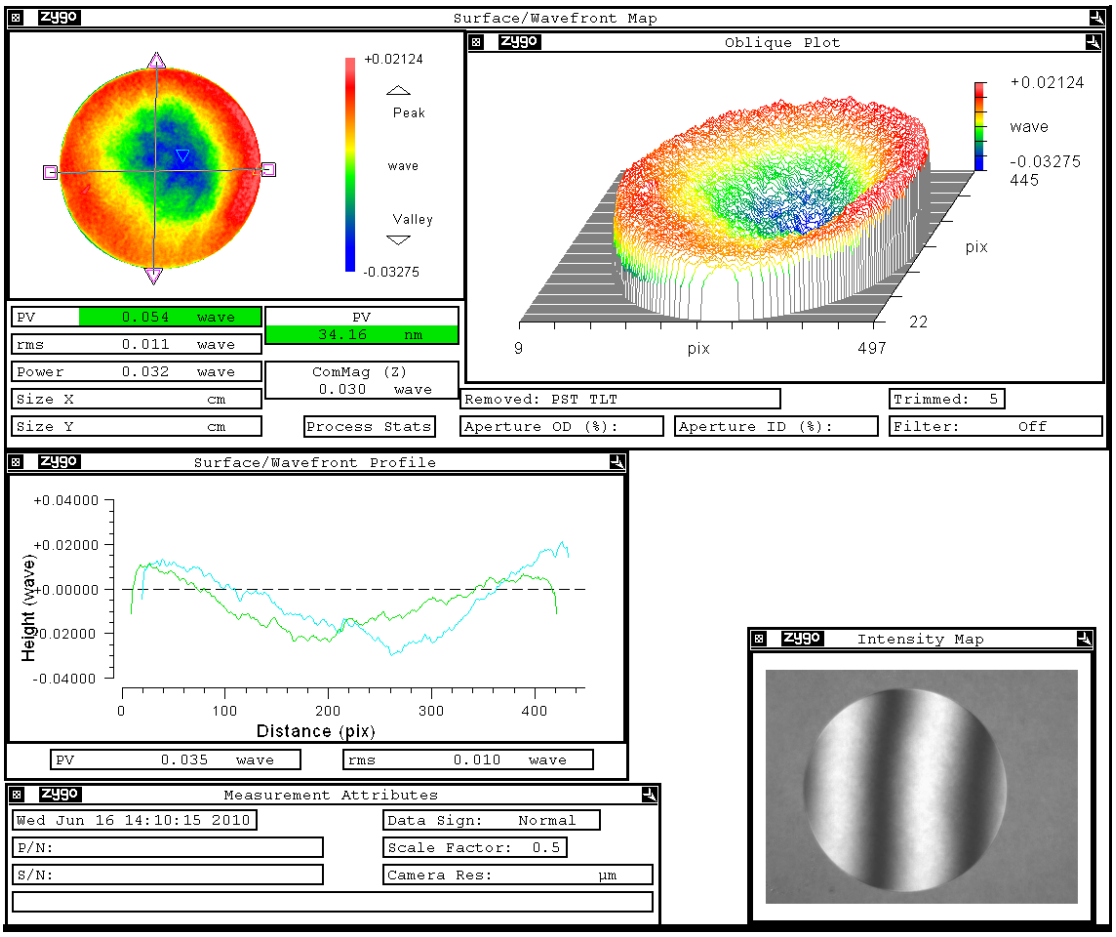

Fig 2.1. An example of the flatness data produced by Zygo interferometer. Flatness is shown in waves and nanometres by default – but can be altered so that all values are shown in nanometres (or other units as desired).

2.2. Cleaning

Each disc was cleaned, in order to optimise bond quality, using the process below.

1. Clean once with cerium oxide.
2. Clean twice with bicarbonate of soda.
3. Rinse with methanol.

4. Wrap in a clean-room cloth until required.

This process removes all marks, dust and residues from the surface of the disc and makes it highly hydrophilic. In this way a good surface for bonding is created.

2.3. Bonding

Bonding solution was prepared using the following steps:

1. Poor 2 ml of sodium silicate solution (commercially available Sigma-Aldrich) and 12 ml of DI water into a centrifuge tube
2. Shake thoroughly
3. Transfer into 1.5 ml centrifuge tubes
4. Centrifuge the solution for 30 seconds at 10000 r.p.m.
5. Transfer into a 0.2 μm medical filter and filter through.

The discs were then bonded using the following process.

1. Wipe the surface of the disc using a clean room cloth and methanol. Check to make sure that the surface is free from marks or dust particles. Repeat for the second disc.
2. Apply 2 μl of the bonding solution by dropping it onto the surface of the first disc with a pipette.
3. Place the second disc on top of the first – it will naturally self-centre, creating a neat bond.

The bonds take just a few minutes to be strong enough to hold the discs in place. However, they do not reach their full strength until four weeks later.

2.4. De-bonding

In the case of bonds twenty-four hours old or younger, de-bonding was performed by rinsing in DI water and applying a slight shearing force. In the case of bonds twenty-four hours old up to forty-eight hours old the samples were de-bonded by placing in a 10% solution of a range of cleaning products in an ultrasonic bath. The products used were; OptiClear, Micro90, Neutracon and Bartoline™ Paint and Varnish Remover, as well as pure DI water, for comparison. Each of these was heated to 40°C. The ultrasonic bath was operating continuously and the samples were checked every twenty minutes. Each time they were checked they were rinsed in DI water for two minutes and a slight shearing force was applied – this occasionally prompted de-bonding.

Where de-bonding was possible the samples were immediately cleaned in the same way as previously. Later, flatness and roughness tests were performed on the de-bonded samples for comparison. Flatness tests were performed using the Zygo interferometer as before, and roughness tests using a VECCO microscope.

3. Results

3.1 Bonding

The process used usually produces a high-quality bond across the full surface of the discs. However, in some cases bubbles become trapped between the discs. Small bubbles are generally pushed to the sides of the discs and expelled, though sometimes

they can remain in the centre. Occasionally bubbles are pushed to the side and remain there – causing a gap in the bond. This greatly reduces its resistance to de-bonding in the ultrasonic bath.

Bonds which contain large gaps at the side of the samples generally de-bond in under two hours. Those which contain smaller bubbles generally de-bond, but tend to take longer. Those without bubbles (or with only central bubbles) are less likely to de-bond.

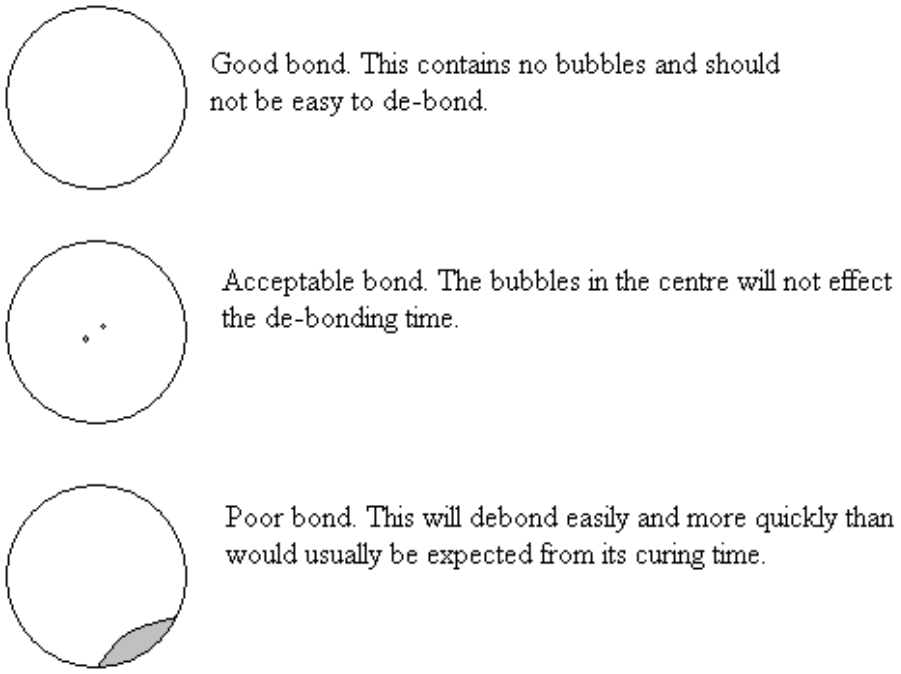

Fig3.1. Possible bonding results and how the de-bonding time will be effected. Bubbles present in the bond do not seem to have much of an effect unless they are very large, or at the edge of the disc.

In cases where the bond was flawed the samples de-bonded more quickly than would be otherwise expected. The percentage of the area of the sample surface that is not bonded is likely to correlate to de-bonding time; however the position of the bubble is likely to be equally important. Central bubbles cannot be reached by the cleaning solution, but bubbles near the edge can be. Also, the imperfect bonds were sketched, and the percentage areas estimated from the sketches. This leads to a high margin of error, in Fig 3.2 and 3.3 the margin of error is estimated as ±2.5%, but it may actually be much larger. In the case of the bonds which were twenty-fours hours old or less, the time is known to the nearest second. The discs which had older bonds, and which were de-bonded in the ultra-sonic bath, were checked every 20 minutes.

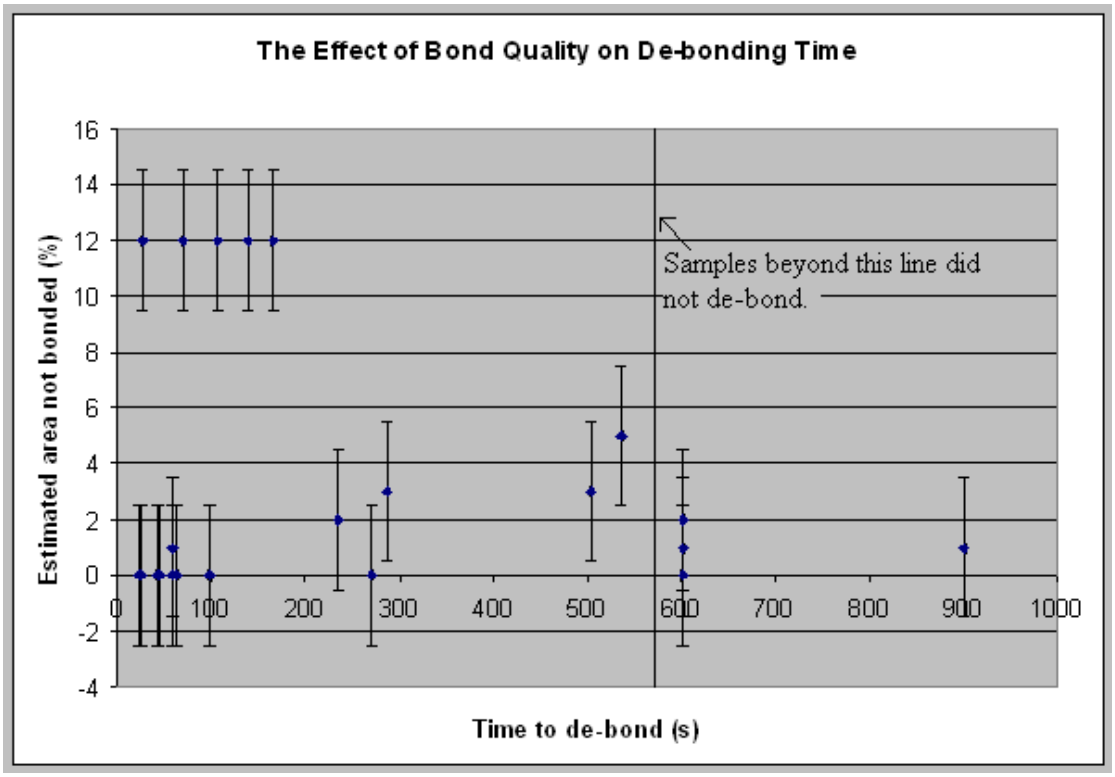

Fig 3.2 The effect on bond quality on de-bonding time for bonds aged up to twenty-four hours. Note time displayed in seconds.

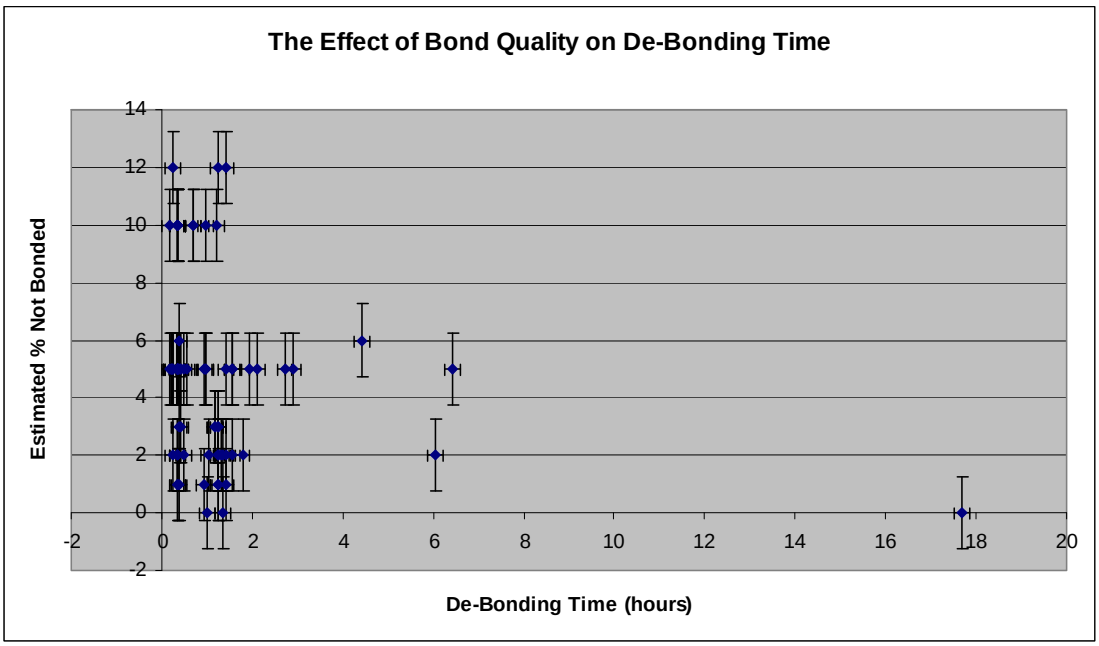

Fig3.3. The effect of bond quality on de-bonding time for bonds aged between twenty-four and forty-eight hours. Note time displaced in hours.

3.2 De-bonding Times.

Figure 3.4 shows a general increase in the time taken to de-bond the samples as the curing time increases. In this case, de-bonding attempts were usually only continued for up to ten minutes (and in one case up to fifteen) to avoid damaging the samples. A

"poor quality bond" is one where 10% or more of the sample is judge not to be properly bonded.

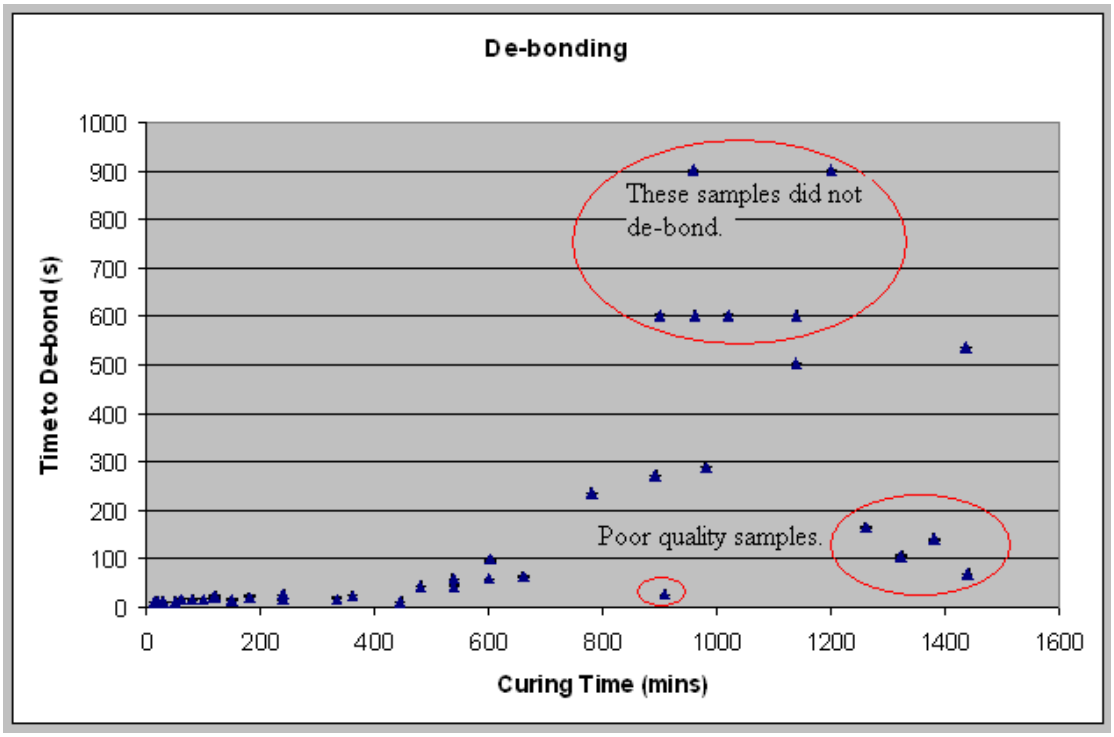

Fig 3.4. The effect of curing time on de-bonding time for samples cured for up to twenty-four hours. The samples that did not de-bond were rinsed for up to fifteen minutes. Samples are considered "poor quality" if more than ten percent of the bonding surface is not bonded.

Figure 3.5 is the same graph again, but with those attempts where the bonds were of poor quality (i.e. those were more than ten percent of the bonding surface is not bonded), having been removed.

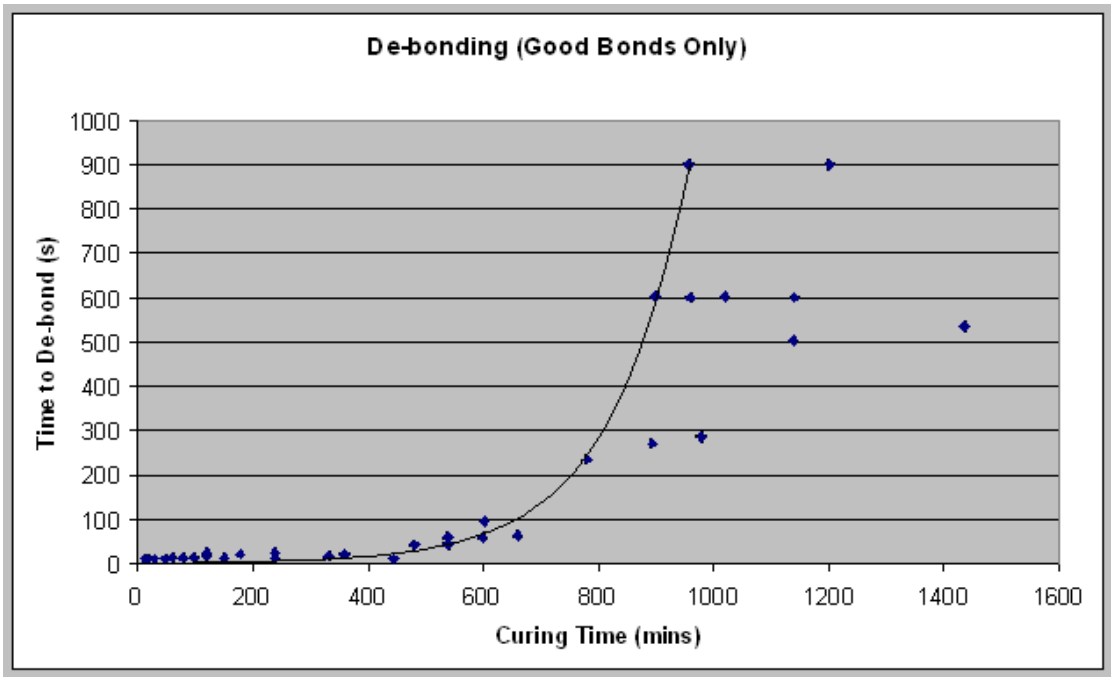

Fig 3.5 The effect of curing time on de-bonding time for samples cured for up to twenty-four hours.

Figure 3.5 demonstrates two points. Firstly, it shows that the oldest bond that was successfully de-bonded in this manner was approximately 23.9 hours old and that all bonds that were cured for thirteen hours or less were successfully de-bonded. Secondly, it shows that the effect of bond quality on de-bonding time can be significant. The curing time in figures 3.4 and 3.5 are known to the nearest minute and the de-bonding time to the nearest second.

Figure 3.6 gives the same relationship again, for bonds cured for between twenty-four and forty-eight hours. In this case the curing time is known to the nearest minute and the de-bonding time to the nearest ten minutes.

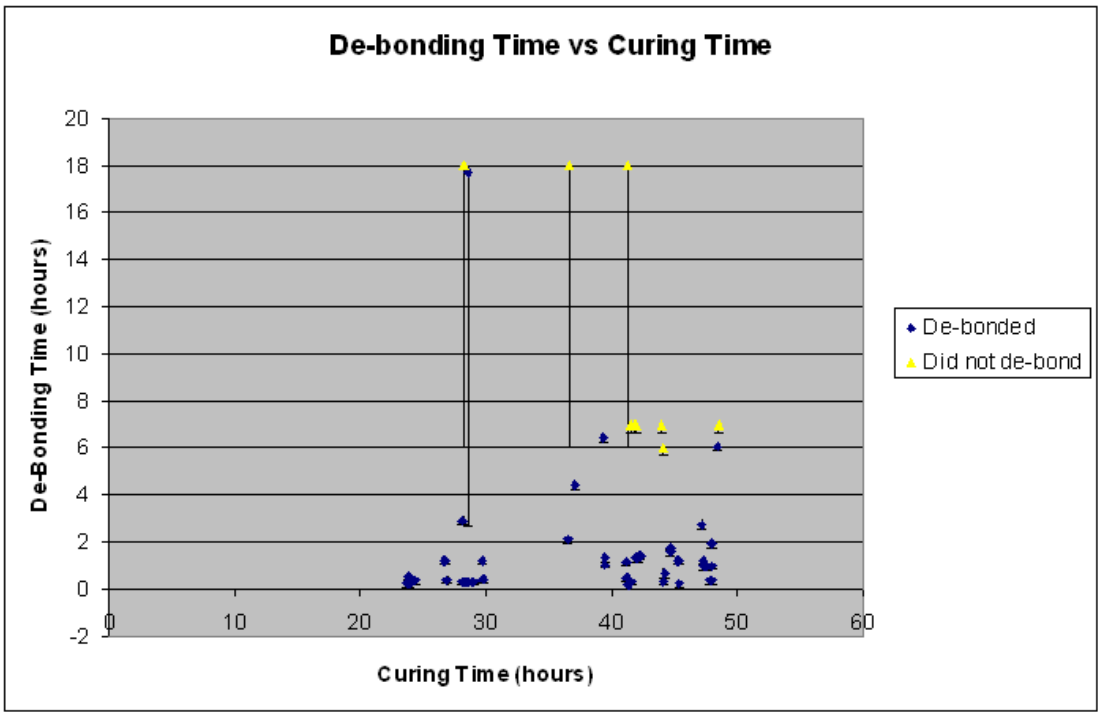

Fig 3.6. Showing the effect of curing time on the length of time required to de-bond samples.

It can be seen that as bonds are cured for longer, more of them fail to de-bond. However, it is also clear that in general the success rate for de-bonding using an ultrasonic bath is very high.

3.3 Temperature and Humidity Effects

The temperature in the laboratory is controlled at 20°C, but varies slightly around this value. Since the temperature was so stable it is not clear whether or not it has an effect on the de-bonding time. During the experiment the maximum recorded temperature 21.8°C and the minimum recorded temperature was 19.8°C. The error on the temperature in this range is ±1%. For temperature, data is used from samples cured for up to forty-eight hours and using both methods of de-bonding.

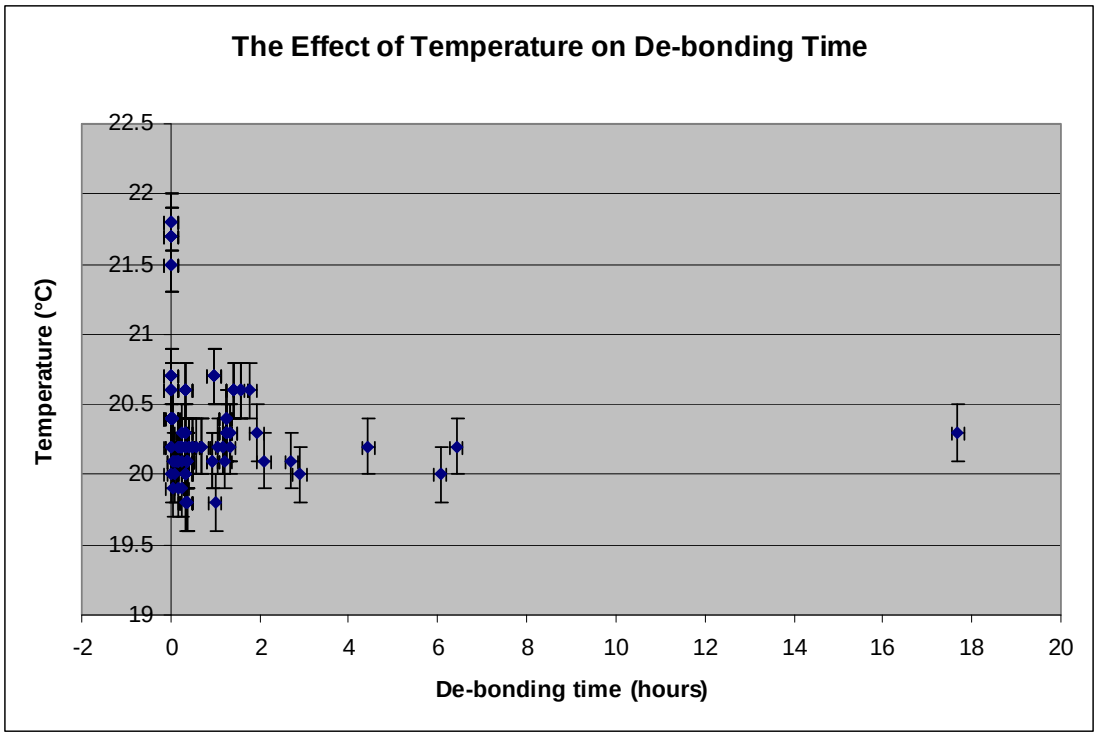

Fig3.7. The effect of temperature on de-bonding time. As can be seen the temperature range was only 2°C and hence it is not clear that there is a relationship between temperature during bonding and the time it takes to de-bond later; although this is likely to be the case.

The humidity, however, is not controlled. As such it varies by a greater degree. During this experiment the minimum humidity RH percentage was 32.9%, the maximum 53.7%. However, it can be seen from Figure 3.8, in this range humidity does not appear to have any great effect. The error in the humidity for humidities below 40% is ±3.5% and above 40% is ±3%. For the graph shown in figure 3.6, all error bars are given as ±3.5%. For humidity effects, data is used from samples cured for up to forty-eight hours and using both methods of de-bonding.

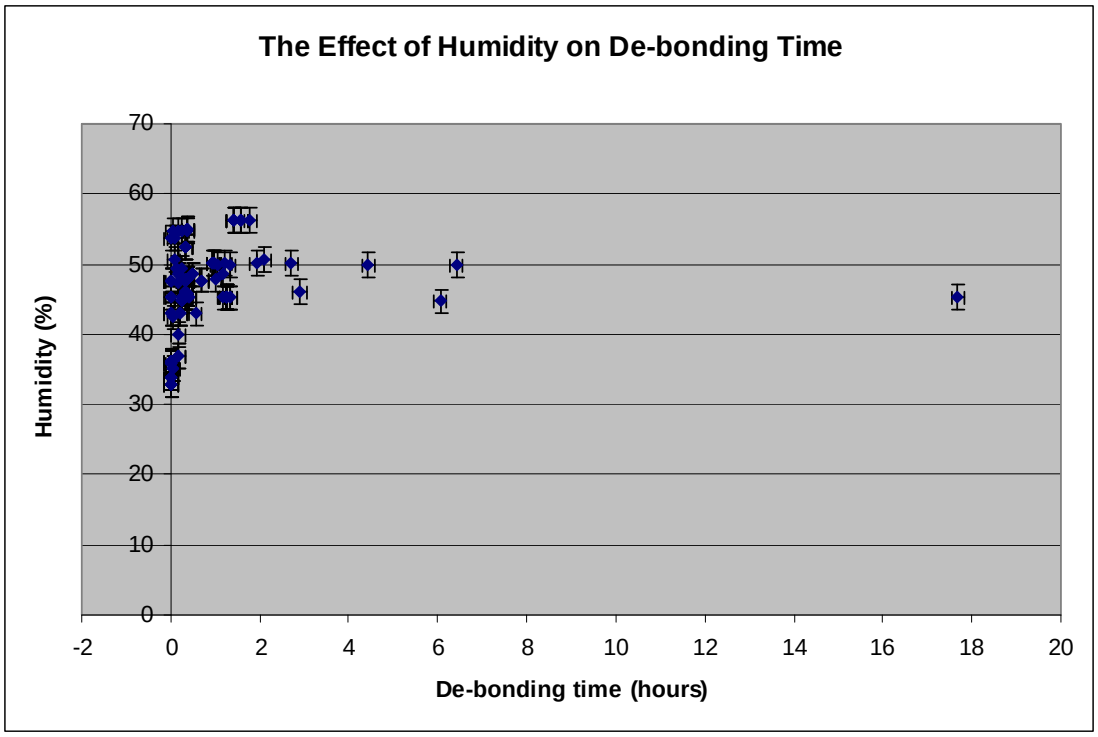

Fig3.6. The effect of humidity on de-bonding time. In this humidity range there appears not to be any relationship between the two values.

3.4 Cleaning Products

Products used to de-bond samples in the ultrasonic bath included OptiClear, Neutracon, Micro90, Bartoline™ Paint and Varnish Stripper and pure DI water, for comparison. The paint stripper was deemed unsafe after a small number of trials and use was discontinued. It begins to boil at 45°C and can cause holes to form in nitrile gloves – without a fume cupboard or more robust gloves the paint stripper should not be used further.

Figure 3.7 compares the de-bonding times for OptiClear, Micro90, Neutracon and water. The times for paint stripper are not shown since only a small number of trials were completed in this manner.

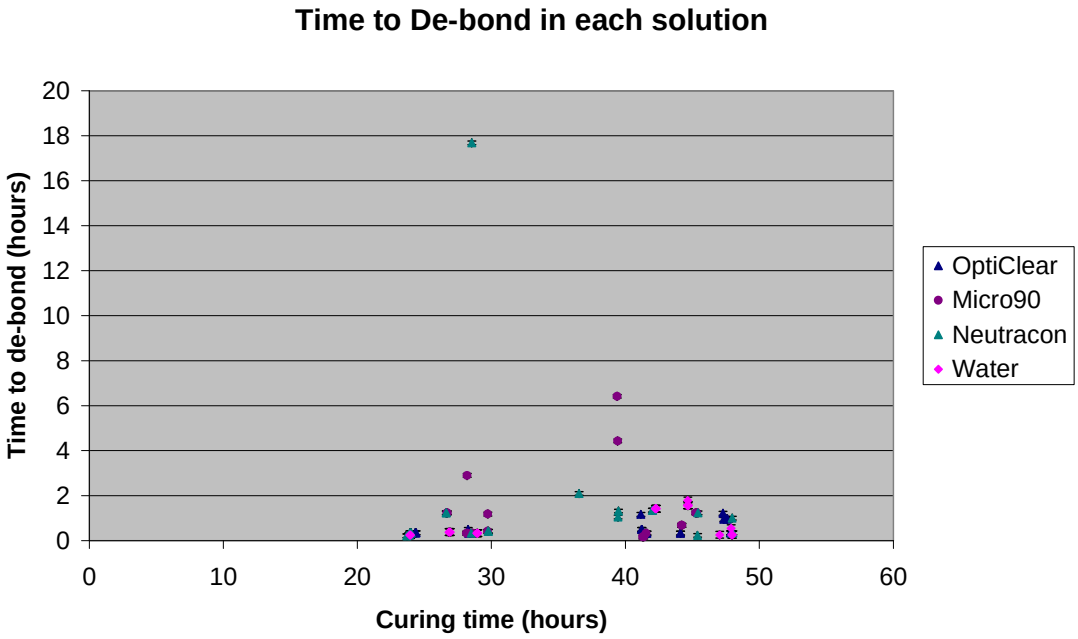

Fig3.7. A comparison in de-bonding time for OptiClear, Micro90 and Neutracon.

Figure 3.8 shows the percentage of samples de-bonded in each solution and in water. Not all samples de-bonded and some solutions de-bonded a greater proportion of samples than others. For example, Neutracon and water both de-bonded one hundred percent of the samples attempted (though Neutracon was used for a greater number of samples); Micro90 de-bonded only 69%.

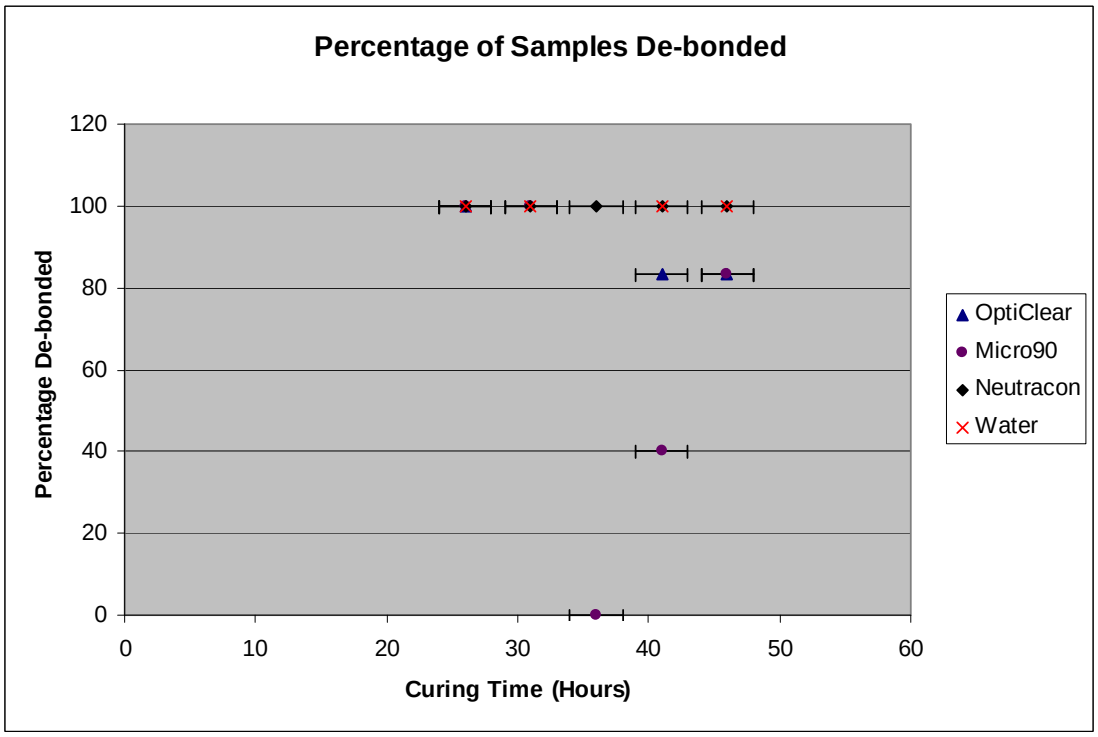

Fig3.8. A comparison of the success rates in de-bonding samples for OptiClear, Micro90, Neutracon and water.

In figure 3.8 the results for Micro90 look somewhat unusual. It should be noted that the thirty-six hour point (where 0% of samples de-bonded) represents only one sample and it that was of a very high bond quality (only 1% was not bonded). Also, at the forty-one hour point, where 40% of the samples de-bonded, 60% had high bond qualities (3% or less of the bonding surface was not bonded). Here five samples were used. It is therefore likely that the odd effect is due to high bond qualities and small numbers of samples.

The success of water and Neutracon for de-bonding is also somewhat surprising. It is possible that this is due to their pH, since both are neutral. However, according to the materials safety data sheets available, OptiClear is also neutral[8]. Micro90 has a pH of 9.5[9] and the pH of the paint stripper is not available.

3.5 Flatness Comparisons

Since not all of the samples were de-bonded, only the data for those which could be de-bonded is available. However, this data is sufficient to demonstrate that the more frequently the samples are bonded and de-bonded, the more their flatness values increase. Figure 3.9 does not show error bars in order to preserve clarity, however, the flatnesses are known to the nearest 4nm.

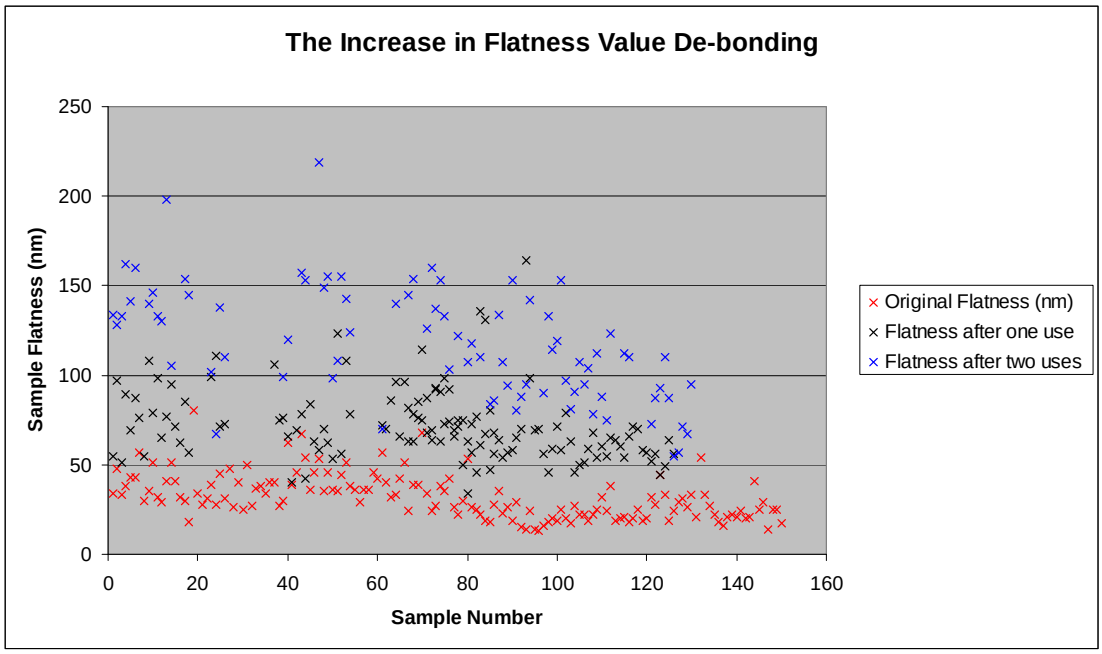

Fig3.9. The effect bonding and de-bonding samples has on their flatnesses. It can be seen that after each trial the flatness value increases.

Where samples that had de-bonded retained a flatness measurement of less than 95 nm they were reserved and re-used. However, it is not practical to use a sample more than twice; only a very small number of samples having been de-bonded twice retained a suitable flatness for re-bonding.

After the samples had been used once, 126 out of the 142 (89%) that de-bonded were flat enough to re-use. After the second use, 20 out of the 87 samples that de-bonded were still flat enough to re-use (23%). However, no sample was used more than twice. None of the samples that had been de-bonded in paint stripper were flat enough to be

re-used, 16% of those de-bonded in Micro90, 36% of those de-bonded in OptiClear, 50% of those de-bonded in Neutracon and 52% of those de-bonded in water were suitable for re-use.

3.5 Disc Roughness

Some interesting features are visible from the roughness tests which are not seen in the flatness tests, and these are worthy of mention. Firstly, in cases where bubbles have been present, the remains of these are occasionally still visible on the surface of the discs. Attempts were made to remove these marks using the cleaning procedure described in section 2.2, but this made no change.

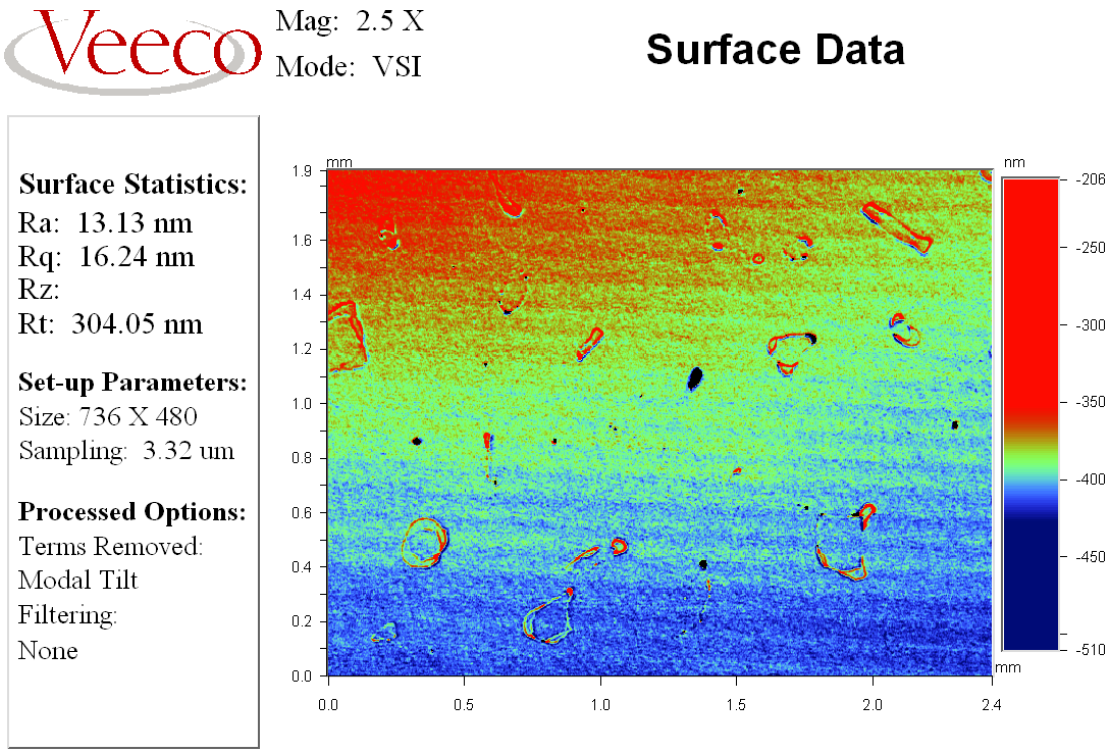

Fig3.10. Marks left behind by bubbles on the surface of a silica disc.

Of the samples used, 73% showed marks on the surface of the discs when roughness measurements were taken. Of these, 82% of the images taken of samples de-bonded in paint stripper, 68% of those de-bonded in OptiClear, 77% of those de-bonded in Micro90, 80% of those de-bonded in Neutracon and 54% of those de-bonded in water showed marks on the surfaces of the discs. These marks generally took the form of small dots or bubbles as can be seen in figure 3.10.

However, in some cases other forms could be seen, for example, the blotch patterns show in figure 3.11 (which are likely to also be left behind by bubbles such as those in figure 3.10). Also note the fern patterns at the edge of some discs, such as those in figure 3.12

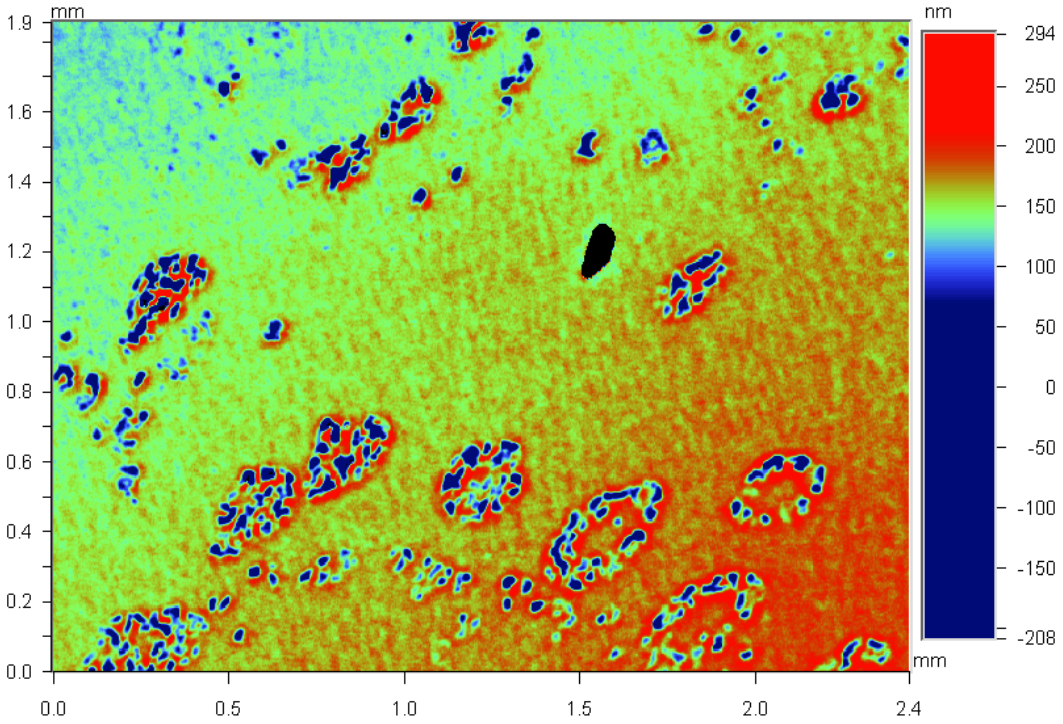

Fig3.11 Blotch patterns on the surface of a disc (x2 magnification).

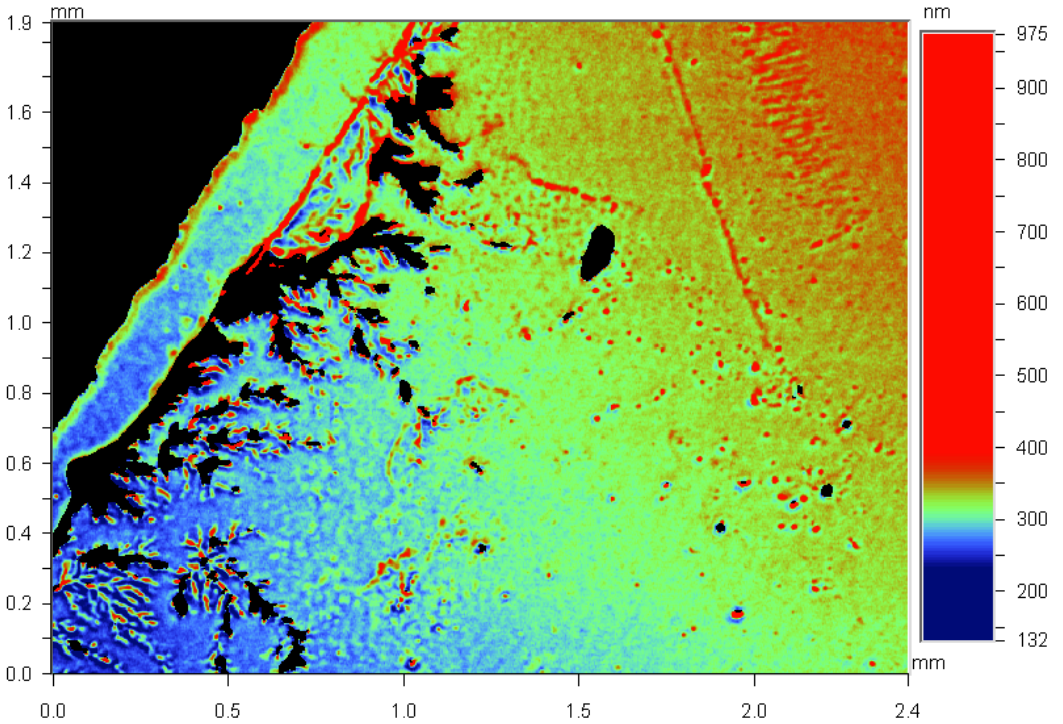

Fig3.12 Fern patterns at the edge of a disc's surface (x2 magnification).

4. Conclusions and Future Work

4.1. Conclusions

It is possible to de-bond samples by rinsing in DI water with bonds up to nineteen hours old. However, it is not easy to do so unless the bonds are imperfect, and may take a long time, in some cases it may not be possible at all. Bonds up to thirteen hours old generally will de-bond in this manner. Younger bonds take less time to de-bond than older bonds.

It is possible to de-bond samples with bonds up to forty-eight hours old in any of the solutions used. Again, it is in general easier and faster to de-bond younger bonds and samples which have imperfect bonds. It is likely that flawed bonds may be de-bonded in this way even when they have been cured for longer than forty-eight hours.

The most promising cleaning product used was Neutracon. This was successful in 100% of the trials it was used for (a total of twenty-three) although in one case this did require leaving the sample in the ultrasonic bath over-night. Samples have been left over-night in other solutions with no success.

Neutracon also has a comparable effect on the disc flatnesses to OptiClear and Micro90. There appears to be little relationship between curing time or de-bonding time and the change in flatness of the discs. However, the discs are almost always less flat after having been de-bonded – as would be expected. Surface damage is occasionally visible, but in most cases this is not so significant that the samples are no longer flat enough to be re-used.

However, pure water also de-bonded all of the samples placed in it (a total of twenty). Additionally, 52% of the samples de-bonded in water were still flat enough to re-use and only 54% of the images take of the surfaces of these samples showed marks or other flaws.

It has been noted that, in the temperature and humidity range in which this experiment was carried out, neither temperature nor humidity have an effect on the de-bonding time. It is possible that either of these would have an effect if a greater range of values were taken into account.

4.2. Future Work

Since pure water and Neutracon seem to have been the most successful at de-bonding samples, further work should be considered in order to find out why, as this is somewhat unexpected. Attempting to de-bond the samples in a greater range of substances (e.g. methanol, acetone, etc) would also be worthwhile.

Up to this point the ultrasonic bath has been used at 40°C, varying this may yield interesting results. Similarly, the solutions used were only 10% with water, altering the concentration should also be considered. Also, the samples were held on tables in beakers within the ultrasonic bath. These place them half-way up the beakers containing the cleaning solution. It may be useful to try other points in the beaker.

Although clearly imperfect bonds are not ideal, it would be useful to know at which point the presence of bubbles produces an effect. The significance of the effect (and how it increases as the percentage area of the surface that is not bonded increases) should also be considered. It is likely that that position of the bubble is as important as the area it covers.

Additionally, whilst the temperature and humidity appeared not to have an effect on de-bonding times during this experiment, it is possible that they would if a greater range were considered. It may be useful to look at this further, to ensure that this de-

bonding method is appropriate for any values of temperature or humidity that Advanced LIGO is likely to have to work at.

5. Acknowledgements

I would like to thank Mariëlle van Veggel for all her help and patience and for teaching me so much so quickly. Also, Nicola Beveridge and Karen Haughian for their help and demonstrations of their work. Prof Jim Hough for the opportunity, Peter Murray for suggesting that I apply and everyone in IGR for being so friendly and welcoming.

6. References

1. http://www.dpf99.library.ucla.edu/session14/barish1412.pdf
2. http://www.phys.ncku.edu.tw/mirrors/physicsfaq/Relativity/GR/grav_speed.html
3. The Science and Detection of Gravitational Waves, Barry B Barish (2002)
4. http://www.nature.com/nphys/journal/v4/n6/full/nphys990.html
5. http://lisa.nasa.gov/
6. http://www.ligo.org/index.php
7. U.S. Utility Patent 6,548,176 – Hydroxide Catalyzed Bonding, Dz-Hung Gwo (2003)
8. http://www.yorketowne.com/MSDS/Chemcraft/431-9150.pdf
9. http://mcf.tamu.edu/docs/msds-pdfs/MICRO-90.pdf

Appendix A – Comments on the Experiments

Occasionally, during bonding and de-bonding studies I made an observation I could not explain. These only occurred rarely, and since I did not know what could have caused them I was unable to repeat my steps to gain more data and investigate further.

Firstly, in many cases when a bond is initially formed bubbles appear. In general, small bubbles are pushed to the sides of the sample and expelled. After one or two hours most samples look perfectly clear, or obviously flawed (in the latter case this can sometimes be spotted quickly enough that they may be de-bonded and the process may be restarted). However, some samples that appeared clear after the first few hours seem to gain bubbles later.

Secondly, when de-bonding samples in the ultra-sonic bath, I observed some of the bubbles in the bonds moving. Large bubbles can break up into many smaller one and vice-versa. In some cases bubbles in the centre move to the edge, and the samples will de-bond shortly afterwards. However, in once case, a bubble moved to the edge of the sample and then vanished. This sample did not de-bond.

Thirdly, on two occasions during de-bonding, one sample slipped partly over the other, and then stuck. This happed on one occasion when de-bonding by rinsing and on one occasion using the ultra-sonic bath. In the both cases I was able to de-bond the samples in approximately two minutes by rinsing in DI water.

I have since noticed that some samples that have not de-bonded were not quite perfectly central, one atop the other. Generally the samples self-centre when they are placed together with the bonding solution. I don't know whether these samples are not central because they didn't do this, or because they slipped later, during de-bonding attempts. The misalignments are very slight, and either case is possible, since it's unlikely that I would have noticed had I not been looking for them.

Finally, not all the samples that contained bubbles later had visible marks left behind from them. I only saw fern patterns in one case, and was unable to find them in any other sample. I don't know what causes either effect.

Appendix B – Additional Work

During my project I also completed a few other smaller pieces of work with the help of others in the IGR group.

With Nicola Beveridge I measured flatnesses, etched and cleaned a set of samples from ingot 3. Later, I was shown the process for acid cleaning these samples, and baking them so that they acquire an oxide layer. Once this was complete I was shown how to use an ellipsometer to measure the thickness of the oxide layer.

The first set of samples I bonded was a set of silica block samples (under the careful instruction of Dr van Veggel). After four weeks of curing, Karen Haughian heat treated these samples – after which I was able to accompany her to observe stress testing.