Mixing ratio 90% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
121	1507.21	1507.21	2.09	9.22	9.93	62.17
122	1774.24	1774.24	2.34	9.12	10.03	72.52
123	1462.76	1462.76	1.81	9.10	10.20	57.94
124	1853.15	1853.15	2.42	9.62	10.15	70.12
125	1521.98	1521.98	2.07	9.16	9.99	62.43
129	1697.85	1697.85	2.12	9.53	10.03	66.41
130	1722.18	1722.18	2.31	9.59	10.17	65.11
131	1599.90	1599.90	2.21	9.54	9.98	63.14
132	1646.11	1646.10	2.26	9.56	10.17	62.43
133	1751.78	1751.78	2.43	9.73	9.98	67.79
134	1859.48	1859.48	2.49	9.39	10.00	74.26
135	1740.03	1740.03	2.30	9.72	9.90	68.49
136	1700.34	1700.34	2.28	9.49	9.96	67.73
Average	**1679.81**	**1679.81**	**2.26**	**9.40**	**10.00**	**66.96**

Table 3.5: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilon Fmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 90% ratio and subjected to cyclic loading.

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Table 3.6 shows the raw data for the group of specimens that were mixed according to the 80% mixing ratio and subjected to fatigue loading.

Mixing ratio 80% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
92	1447.52	1447.52	2.52	7.35	10.02	73.56
93	1601.98	1601.98	2.77	7.84	9.81	79.62
94	1220.89	1220.89	1.83	8.53	10.03	53.35
95	1356.86	1356.86	2.11	8.35	9.86	62.68
96	1530.53	1492.32	2.49	7.95	9.91	73.51
97	1858.05	1858.05	3.04	8.67	10.07	79.25
98	1454.38	1454.38	2.58	8.21	9.81	69.03
99	1971.04	1971.04	2.95	9.27	10.06	78.79
100	1454.14	1454.14	2.18	9.27	10.06	58.12
101	1754.26	1754.26	2.60	8.67	10.09	74.53

Mixing ratio 80% - fatigued						
specimen Nr	Fmax N	Fbreak N	Epsilon-Fmax mm	Width b mm	Height d mm	FS MPa
102	1783.76	1783.76	2.48	9.12	10.12	71.62
103	1824.90	1824.90	2.54	9.37	9.98	73.33
104	1522.18	1522.18	2.83	7.63	10.01	74.66
105	1277.98	1277.98	1.94	8.17	10.07	57.85
106	1408.02	1408.02	2.12	8.36	10.05	62.53
107	1422.44	1422.44	2.11	8.75	9.89	62.33
108	1662.53	1662.53	2.55	8.65	10.06	71.22
109	1647.65	1647.65	2.97	8.04	9.82	79.69
110	1486.74	1486.74	1.99	9.53	9.96	58.97
111	1787.61	1787.61	3.07	8.85	9.81	78.71
112	1808.88	1808.88	2.72	9.50	9.84	73.74
113	1602.76	1602.76	2.70	7.59	10.10	77.63
Average	**1585.69**	**1583.95**	**2.50**	**8.53**	**9.97**	**70.21**

Table 3.6: Highest load measured (Fmax), load at failure (Fbreak), deflection (epsilon Fmax), width (b); height (d) and flexural strength (FS) for the specimens mixed according to the 80% ratio and subjected to cyclic loading.

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3.2.4 Descriptive statistics

The following analysis presents the descriptive statistics for the different mixing ratio values (80%, 90%, 100%) and fatigue level (1=fatigued, 0=not fatigued): the highest force registered before failure of the specimens (Fmax in N), the force registered at fracture (Fbreak in N), the width and height of the specimens (mm), deflexion (epsilon in mm), and the calculated flexural strength (FS in MPa).

Table 3.7 gives a summary of statistics for each mix Ratio-Fatigue combination.

Mix	Fatigued	# Obs	Variable	Mean	Median	Std Dev	Minimum	Maximum
80%	0	24	Fmax	1467.75	1519.80	265.77	639.58	1811.33
			Fbreak	1467.75	1519.80	265.77	639.58	1811.33
			Epsilon	2.32	2.36	0.44	0.99	2.82
			Width	8.35	8.29	0.41	7.59	9.08
			Height	10.01	10.00	0.11	9.84	10.19
			FS	66.05	68.44	12.28	27.13	80.60
	1	22	Fmax	1585.69	1566.26	202.05	1220.89	1971.04

Mix	Fatigued	# Obs	Variable	Mean	Median	Std Dev	Minimum	Maximum
			Fbreak	1583.95	1562.08	202.71	1220.89	1971.04
			Epsilon	2.50	2.55	0.37	1.83	3.07
			Width	8.53	8.59	0.64	7.35	9.53
			Height	9.97	10.02	0.11	9.81	10.12
			FS	70.21	73.42	8.27	53.35	79.69
90%	0	22	Fmax	1548.18	1582.13	299.29	1005.18	1951.48
			Fbreak	1543.61	1582.13	297.36	1005.18	1943.44
			Epsilon	1.98	2.00	0.45	1.16	2.68
			Width	9.40	9.41	0.28	8.90	9.84
			Height	10.12	10.15	0.10	9.93	10.29
			FS	60.41	61.86	11.83	38.89	76.56
	1	20	Fmax	1679.81	1711.26	145.50	1462.76	1964.43
			Fbreak	1679.81	1711.26	145.50	1462.76	1964.43
			Epsilon	2.26	2.29	0.22	1.81	2.71
			Width	9.40	9.44	0.22	8.93	9.73
			Height	10.00	10.00	0.10	9.84	10.20
			FS	66.96	67.07	5.66	57.94	80.95
100%	0	21	Fmax	1465.99	1592.93	289.05	781.54	1848.77
			Fbreak	1463.40	1562.90	287.88	781.54	1848.77
			Epsilon	1.91	1.99	0.42	0.97	2.46
			Width	9.40	9.38	0.35	8.31	9.81
			Height	10.13	10.11	0.12	9.90	10.33
			FS	56.92	58.73	10.78	31.27	71.14
	1	19	Fmax	1665.38	1682.69	155.06	1337.31	1984.88
			Fbreak	1663.83	1682.69	156.75	1337.31	1984.88
			Epsilon	2.29	2.29	0.23	1.80	2.80
			Width	9.56	9.42	0.45	8.85	10.56
			Height	10.08	10.06	0.17	9.89	10.58
			FS	64.29	63.94	5.16	52.06	73.19

Table 3.7: Summary of the statistics for each Mix ratio-Fatigue combination. 0= not fatigued; 1= fatigued; FS= flexural strength

3.2.5 Analytical statistics

3.2.5.1 Fmax

Table 3.8. Shows a summary of the descriptive statistics for Fmax for all the groups.

groups	n	min	max	mean	median	std dev.
100% (0)	21	781.54	1848.77	1465.99	1592.93	289.05
100% (1)	19	1337.31	1984.88	1663.83	1682.69	155.06
90% (0)	22	1005.18	1951.48	1548.18	1582.13	299.29
90% (1)	22	1462.76	1964.43	1679.81	1711.26	145.50
80% (0)	24	639.58	1811.33	1467.75	1519.80	265.77
80% (1)	22	1220.89	1971.04	1585.69	1566.26	202.05

Table 3.8: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (STD dev) of the highest load measured in newton (Fmax). (0= no fatiguing; 1= fatiguing)

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Initially a comparison was drawn between the mean Fmax and means Fmax values for the different ratio and fatigue groups.

For the same mixing ratio, the mean, as well as the median Fmax after fatiguing (1) is always higher than the mean and the median Fmax of the groups that were not fatigued (0).

For both fatigued and unfatigued groups, the Fmax increases from 80% group to 90% group. The mean difference in Fmax between the mixratio of 80 and the mixratio of 90, over both fatigued and not fatigued, is only very marginally significant (P=0.087 for both groups) ($p<0.05$) When comparing the median values for mixratio 90%-100% though, the trend of Fmax values for the fatigued specimens is essentially in the opposite direction.

A graphical representation of the distribution of Fmax defined by factors of fatigue and ratio is given by a boxplot (Figure 3.3).

The ends of the box are approximate quartiles and the heavy line in the middle is the median. The table of means shows similar trends, as do the (analysis of variance) significance tests which are essentially comparisons of means. The (main effect), Fatigue is statistically significant, P=0.001.

Further examination of the dimensions of the specimens was done to possibly explain this trend fully.

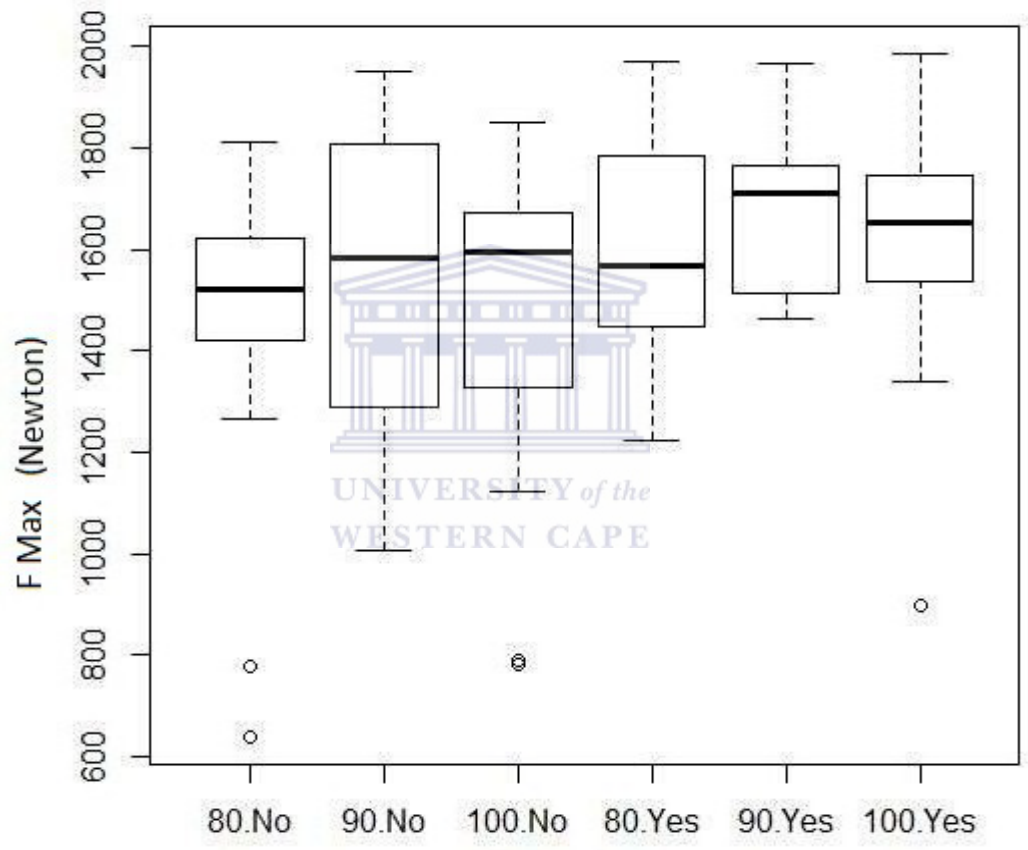

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Figure 3.3: Box and whiskers plot of the median Fmax for the 3 mixing ratios without cyclic loading (no) and with cyclic loading (yes).

3.2.5.2 Dimensions of the specimens

After removal from the mould, the trapezoid specimens were machined and thereafter finished off by hand into square shaped blocks. There could, therefore, be small fluctuations in width and height of the specimens. Consequently each specimen was carefully measured in width and height.

3.2.5.2.1 Width

Table 3.9 shows a summary of the descriptive statistics for width for all the groups.

groups	n	min	max	mean	median	std dev
100% (0)	21	8.31	9.81	9.40	9.38	0.35
100% (1)	19	8.85	10.56	9.56	9.42	0.45
90% (0)	22	8.90	9.84	9.40	9.41	0.28
90% (1)	22	8.97	9.73	9.40	9.44	0.22
80% (0)	24	7.59	9.08	8.35	8.29	0.41
80% (1)	22	7.35	9.53	8.53	8.59	0.64

Table 3.9: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (std dev) of the width measured in mm. (0= no fatiguing; 1= fatiguing)

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The mean width of the different groups was compared using a 2-way ANOVA test of fixed effects (Table 3.10). The numerator degrees of freedom and the denominator degrees of freedom are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	85.02	< .0001
fatigued	1	122	2.44	0.12
mix*fatigued	2	122	0.58	0.56

Table 3.10: ANOVA test of fixed effects for the widths. Numerator (Num),Denominator (Den), Degrees of Freedom (DF).

Table 3.11 shows an abbreviated version of the Least Squares Means test. * Pairwise comparisons show the mean width for 80 is significantly lower than the mean width for 90 or 100 ($p<0.0001$).

mix	Estimate	Standard Error
80*	8.44	0.062
90	9.4	0.065
100	9.48	0.066
Fatigue	Estimate	Standard Error
0 (No)	9.05	0.051
1 (Yes)	9.17	0.054

* = statistically significant

Table 3.11: Statistical evidence of width differences using a least squares means test.

A box and whisker plot was used to demonstrate the different values and their effects on the distribution of the width of specimens (Figure 3.4).

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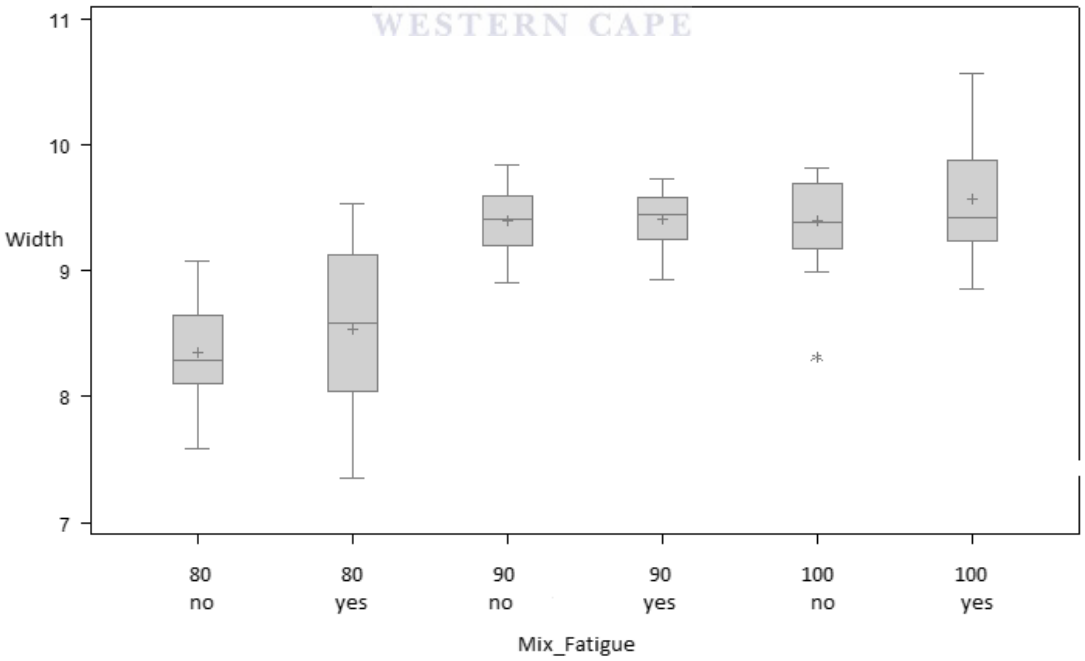

Figure 3.4: Boxplot of width for groups defined by the factors, fatigue and mixing ratio. The ends of the box are approximate quartiles and the line in the middle is the median. The + sign in the box represents the mean.

When considering width, there is a significant difference between that for 80% and each of 90% and 100% groups.

3.2.5.2.2 Height

Table 3.12 shows a summary of the descriptive statistics for height for all the groups.

groups	n	min	max	mean	median	st dev
100% (0)	21	9.90	10.33	10.13	10.11	0.12
100% (1)	19	9.89	10.58	10.08	10.06	0.17
90% (0)	22	9.93	10.29	10.12	10.15	0.10
90% (1)	22	9.84	10.20	10.00	10.00	0.10
80% (0)	24	9.84	10.19	10.01	10.00	0.11
80% (1)	22	9.81	10.12	9.97	10.02	0.11

Table 3.12: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the height measured in mm. (0= no fatiguing; 1= fatiguing)

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Similarly Table 3.13 shows statistical evidence of height differences using ANOVA test of fixed effects . The numerator degrees of freedom and the denominator degrees of freedom are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	10.05	< .0001
fatigued	1	122	10.18	0.0018
mix*fatigued	2	122	1.39	0.2533

Table 3.13: ANOVA test of fixed effects. Numerator(Num),Denominator(Den), Degrees of Freedom(DF).

Table 3.14 shows an abbreviated version of the Least Squares Means test for the height of the specimens. * Pairwise comparisons show the mean for 80 is significantly lower than the mean for 90 or100 ($p<0.0001$)

mix	Estimate	Standard Error
80*	9.99	0.017
90	10.06	0.018
100	10.11	0.019

Fatigue	Estimate	Standard Error
0 (No)	10.08	0.01
1 (Yes)	10.02	0.02

* = statistically significant

Table 3.14: Statistical evidence of height differences using a least squares means test.

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A box and whisker plot was used to demonstrate the different values and their effects on the distribution of the height of specimens. Figure 3.5 illustrates this clearly.

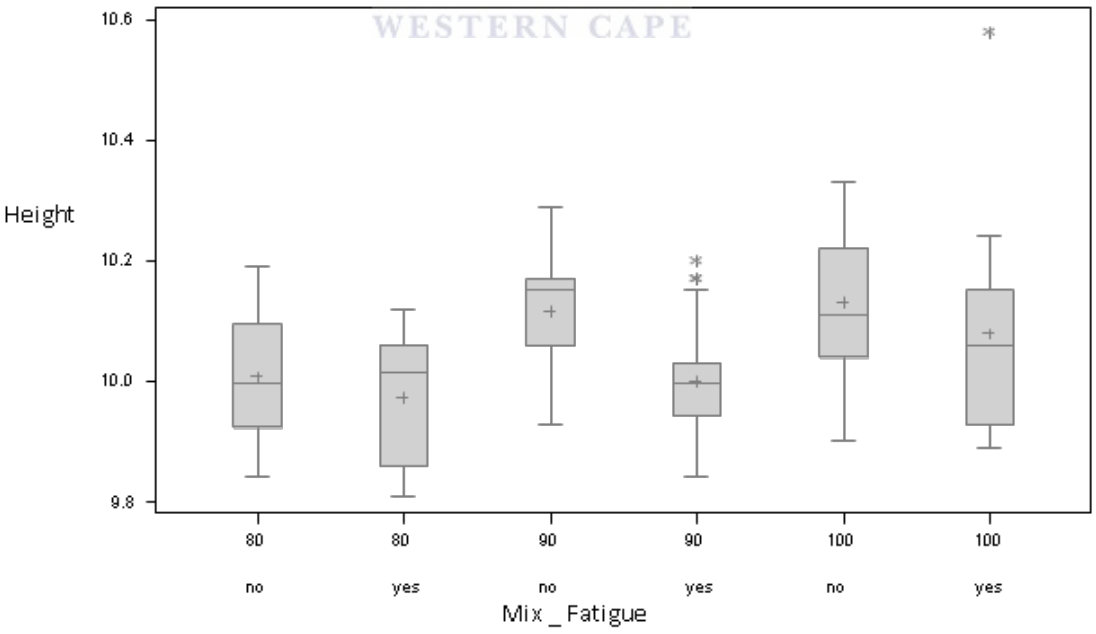

Figure 3.5: Boxplot of height for groups defined by the factors, fatigue and mixing ratio. The ends of the box are approximate quartiles and the line in the middle is the median. The + sign in the box represents the mean.

For height there is one outlier in the data (a value of 10.58 for the group with 100%, with fatiguing). With, or without the outlier analysis indicates significant differences in height.

As further confirmation of the influence of height and width on the properties of our specimens Figure 3.6 shows the distribution of thicklr (height and width and length) in the various mixing ratios and fatigue subgroups. Every dot represents one observation.

It is noted that the 80% 'yes' and 'no' values do not even overlap. This confirms the finding that the 80% group varies considerably from the 90% and the 100% groups in width and height.

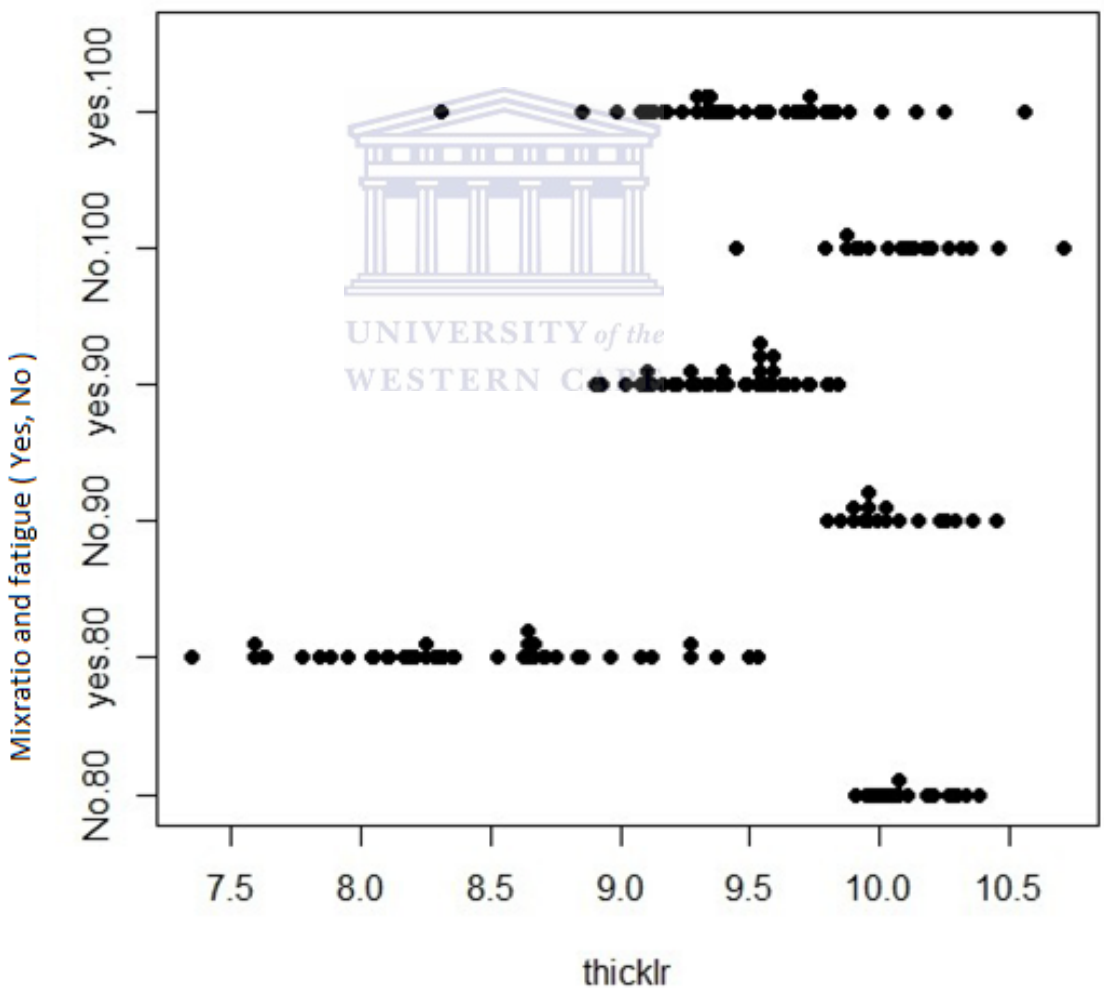

Figure 3.6: The distribution of thicklr (height and width and length) in the various mixratio and fatigue subgroups. Every dot represents one observation.

3.2.5.3 Deflexion

A measure of deflexion is given by the epsilon values before failure of the different specimen groups.

Table 3.15 shows a summary of the descriptive statistics for deflexion (epsilon) for all the groups.

groups	n	min	max	mean	median	sd
100% (0)	21	0.97	2.46	1.91	1.99	0.42
100% (1)	19	1.80	2.80	2.29	2.29	0.23
90% (0)	22	1.16	2.68	1.98	2.00	0.45
90% (1)	22	1.81	2.71	2.26	2.29	0.22
80% (0)	24	0.99	2.82	2.32	2.36	0.44
80% (1)	22	1.83	3.07	2.50	2.55	0.37

Table 3.15: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the deflexion measured in mm. (0= no fatiguing; 1= fatiguing)

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The results and plot of the residuals indicates skewness and non-normality. For this reason a nonparametric approach was taken rather that the more commonly used Kruskal-Wallis or Friedman tests. As the data was multifactorial a method known as the Aligned Ranks Transform (ART) was used for the analysis (Mansouri, 1999). The ART analysis tool was used to align and rank the data. ANOVA analysis was then done. The software used for statistical analysis was SAS v9 (SAS Institute Inc., Cary, NC, USA). The ART analysis was done in SAS using a user constructed macro.

Table 3.16 shows the results of the ART for the variable Epsilon. Numerator(Num), Denominator(Den), Degrees of Freedom(DF). The Num DF and the Den DF are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
mix	2	122	12.46	< .0001
fatigued	1	122	15.39	0.0001
mix*fatigued	2	122	0.56	0.5725

Table 3.16: Statistics of a nonparametric ART analysis of the data for the variable Epsilon.

Table 3.17: Shows a summary of the statistics of least squares means for the variable Epsilon. * Pairwise comparisons show the mean width for 80 is significantly lower than the mean width for 90 or 100 ($p<0.0001$). ** Mean with Fatigue significantly higher than without fatigue ($p=0.0001$)

mix	Estimate	Standard Error
80*	2.42	0.055
90	2.12	0.057
100	2.1	0.059
Fatigue	Estimate	Standard Error
0 (No)**	2.07	0.046
1 (Yes)	2.35	0.048

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Table 3.17: Summary of the statistics of least squares means for the variable Epsilon.

Figure 3.7 shows boxplots of deflexion (using the epsilon values for strain development) against the mixing ratios and fatigue subgroups. The trends indicated by this graph are that median deflexion decreases from 80% to 90% groups and remains stable thereafter. A two way analysis of variance with response variable deflex and factors Mixratio and Fatigue confirms that there is a statistically significant change from 80% to 90%, ($p<0.001$), and that the change from 90% to 100% groups is not significant. Main effect Fatigue is significant, $p<0.001$.

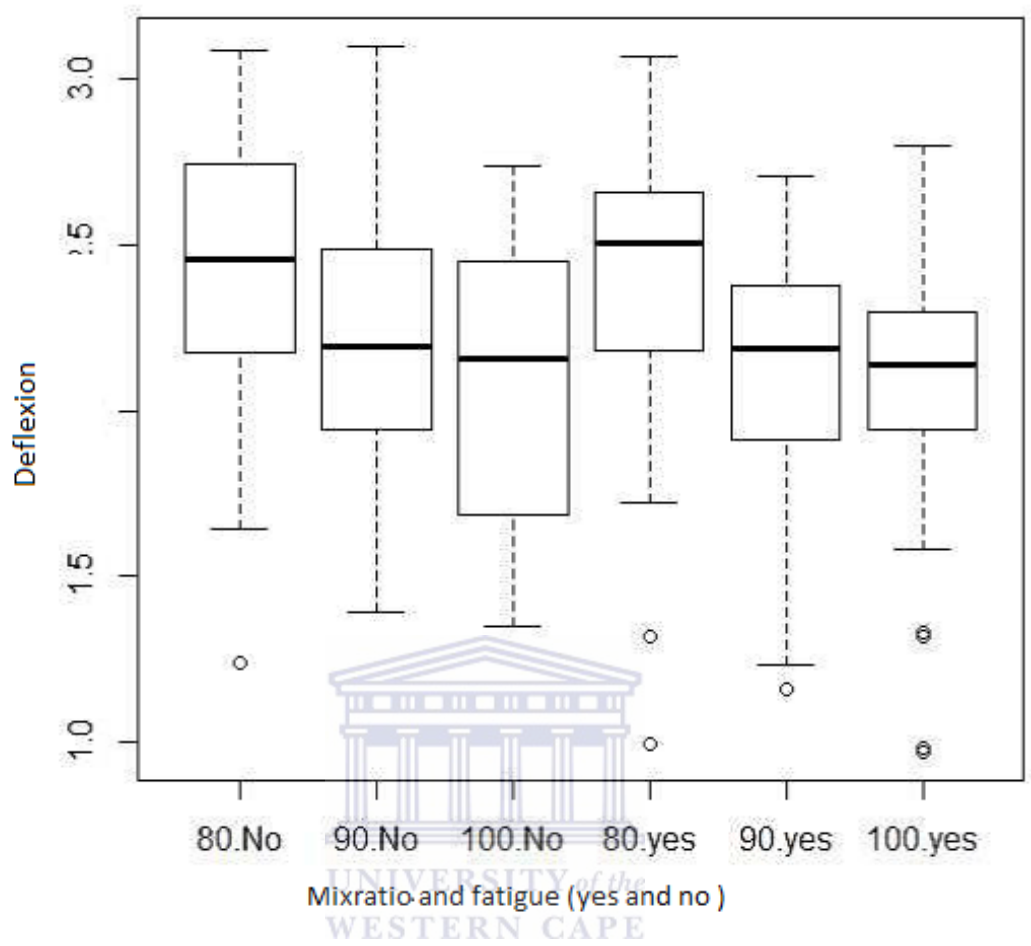

Figure 3.7: Boxplots of deflexion (epsilon Fmax) for the mixratio x fatigue subgroups.

3.2.5.4 Flexural strength

Table 3.18 shows a summary of the descriptive statistics for flexural strength (FS) for all the groups.

groups	n	min	max	mean	median	sd
100% (0)	21	31.27	71.14	56.92	58.73	10.78
100% (1)	19	52.06	73.19	64.29	63.94	5.16
90% (0)	22	38.89	76.56	60.41	61.86	11.83
90% (1)	22	57.94	80.95	66.96	67.07	5.66

groups	n	min	max	mean	median	sd
80% (0)	24	27.13	80.60	66.05	68.44	12.28
80% (1)	22	53.35	79.69	70.21	73.42	8.27

Table 3.18: Minimum value (min), maximum value (max), mean (mean), median value (median) and standard deviation (sd) of the flexural strength measured in MPa. (0= no fatiguing; 1= fatiguing)

The variation in width, height and deflexion confirms that the standardized variable of FS would be appropriate to be used for analysis. Flexural strength was calculated using the equation: $FS= 3FMaxL/2bd^2$

Initially a standard two-way analysis of variance was done. However examination of the residuals from the model indicates that they are not normally distributed. For this reason a nonparametric approach was taken. The ART was again used. These analyses demonstrate that there is no significant interaction between Mix and Fatigue state, that the 80 mix has a significantly higher mean than either the 90% or 100% groups (with differences of about 4.4 and 7.5 units respectively), and that the Fatigued state has a higher mean than the Not Fatigued state by about 6.0 units.

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Table 3.19 shows the results of a nonparametric ART analysisfor the variable FS. Numerator (Num), Denominator (Den), Degrees of Freedom (DF). The Num DF and the Den DF are considered parameters of the test statistic. The test statistic follows an F distribution. 'F Value' is the value of the test statistic. 'Pr>F'gives the probability of getting a value of the F statistic that is larger than the one observed. It is the p-value for the test.

	Num	Den		
Effect	DF	DF	F Value	Pr > F
Mix	2	122	11.82	< .0001
Fatigued	1	122	10.53	0.0015
mix*fatigued	2	122	0.26	0.7707

Table 3.19 Results of the ART for the variable FS.

Table 3.20: Shows a summary of the statistics of a nonparametric ART analysis of the data for least squares means for FS. * Pairwise comparisons based on ART analysis show the mean FS for 80% is significantly lower than the mean FS for 90% ($p<0.0001$) or 100% ($p=0.0026$)

** Mean FS with Fatigue significantly higher than without ($p=0.0015$)

mix	Estimate	Standard Error
80*	68.13	1.42
90	63.68	1.48
100	60.6	1.52

Fatigue	Estimate	Standard Error
0 (No)**	61.12	1.18
1 (Yes)	67.15	1.23

Table 3.20: Results of the ART analysis of the data for least squares means (FS)

In Table 3.21 both the fatigued and unfatigued groups display an increasing flexural strength. The 80% group in both fatigued and unfatigued specimens have the highest FS.

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Unfatigued					
Mixing ratio	**F Max in N**	**Deflexion**	**Width**	**Thickness**	**Flexural strength**
100%	1557.85	2.04333	9.40444	10.13	60.6221
90%	1674.81	2.16823	9.39588	10.11	65.5972
80%	1536.71	2.43	8.32045	10	69.3206
Fatigued					
Mixing ratio	**F Max in N**	**Deflexion**	**Width**	**Thickness**	**Flexural strength**
100%	1665.38	2.2879	9.56421	10.08	64.2886
90%	1679.81	2.261	9.4045	10	66.9615
80%	1585.69	2.504	8.53045	9.9741	70.2145

Table 3.21: Flexural strength (MPa) defined by mixratio and fatigue status and sample dimensions.

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CHAPTER 4

Discussion

4.1 Introduction

The aim of this research study was to investigate the influence of changing P/L ratios on the fatigue behavior of a fibre reinforced PMMA used for denture bases.

The FS of 3 different P/L ratios of fibre-reinforced PMMA was compared. Half of the specimens in each P/L group were subjected to fatigue loading before the 3-point bending test was done.

The median and mean FS values before and after cyclic loading were calculated and compared by means of a non-parametric analysis of variance (ART). A p-value of less than 0.05 was considered significant.

The original protocol of this study accepted certain outcomes i.e.: a certain degree of adhesion between fibre and matrix. This proved to be wrong. The results from this study were unexpected and therefore it is difficult to answer the original research question.

Besides the unexpected adhesion problem, the researcher was faced with 2 further major challenges. The extent of these challenges could not be anticipated during the development of the protocol. The first was the difficulty of the manufacturing of the specimens using a custom-made template. This could not be deduced from reading literature on similar research projects. The second one was the infrastructure and expertise necessary to do cyclic loading.

Therefore, this discussion will start with a presentation of the piloting process prior to the discussion of the results.

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4.2 Piloting process

4.2.1 The mould

The mould was designed to fit the length of the donated ever Stick fibres and to mimic the thickness of a denture base as closely as possible (Jerolimov *et al.,* 1989, Kanie *et al.,* 2000, Bertassoni *et al.,* 2008). An attempt was made to position the fibres closer to one side of the specimens to make full use of the strength supplied by inner support on the tension side of a material (Narva *et al.,* 2005b). It seemed almost impossible to position the fibres on the tension side of the these thin (4mm) specimens. The doughy consistency of the heat cured PMMA made this impossible. Researchers in previous studies pre-wet their fibres to obtain a better bond between fibre and PMMA (Vallittu 1999, Tacir 2006).

A few specimens were manufactured and sent to the CSIR in Pretoria for pilot testing (See 4.2.4: Cyclic loading). CSIR established that the fibres did not lie reliably on the tension side of the specimens and made results unreliable. The height of the specimens was to be increased by 6 mm to a total of 10 mm of height to facilitate correct positioning of the fibre bundle and calibration of the testing equipment. The depth of the cavities in the mould was modified accordingly.

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4.2.2 The fibres

The research proposal was sent to the manufacturers of the fibres in Finland for possible sponsoring of the fibres. They kindly agreed. The fibres suggested by the Stick Company in Finland were 50mm pre-impregnated ever Stick fibres (Stick Tech). Consequently, 150 fibres for the complete project were donated by the manufacturers.

Due to the high cost of the fibres, the manufacturing of the specimens was extensively piloted using metal wires and resin impregnated superfloss as substitutes for the fibre. Both the wire and PMMA impregnated superfloss behaved differently from the fibres that were subsequently used. The fibres were much more difficult to work with.

The fibres were not all of equal length, some being longer, some shorter than the 50mm as stated in the marketing brochure. This complicated the accurate manipulation of the fibers and therefore complicated the manufacturing of the specimens. The custom-made mould

was already fabricated and the ends did not reach the slots. For these shorter fibres, an additional method of stabilization of the fibre was designed in the form of a staple-like thin metal wire (Figure 2.3 Methodology). The longer fibres were cut with surgical scissors to be exactly 50mm long (Figure 2.4: Methodology). The small light curing oven used for special tray manufacture was initially tried for the polymerization that the manufacturers required. Finally the pre-impregnated fibres were light polymerized for 2 minutes using a curing light (Megalight Mini, Radeburg, Germany).

4.2.3 Manufacturing of the specimens

In order to adhere to the protocol and limit unnecessary variations in specimen design and compilation manufacturer's instructions had to be followed carefully. These instructions demanded that the PMMA is mixed and left to reach the dough stage before packing inside the mould. The dough consistently pushed the fibers from their correct position when the mould was closed and pressure was applied (Figure 2.5: Methodology). A problem such as this one has not been mentioned in the literature of similar studies (Dogan et al., 2007; Bertassoni *et al.*, 2008; Fajardo *et al.*, 2011). At first it was attempted to close the mould lid extremely slowly (22 minutes per closure) so as not to dislodge the fibres. This did not work.

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The solution was finally to manufacture the specimens in 2 stages. The cavities were filled with a first layer of PMMA-dough up to the level of the slots where the ends of the fibre bundles were to be positioned. The original protocol described a single stage procedure with a flat cover. This proof-packing required an additional mould cover to be made. This additional cover was made with platforms that protruded into the cavities of the base up to the level of the stops (Figure 2.1: Methodology).

A second mixture using the same P/L ratio was mixed and immediately poured over the fibres to overfill the cavities (Figure 2.8: Methodology). This second layer was left to "dough" in situ for the required 15 minutes before slowly closing the mould with the flat cover and applying pressure.

The mould was then compressed in a laboratory press (Figure 2.9: Methodology). As before, to avoid the risk of displacing the fibres, the press was closed very slowly. Every batch was polymerized for 20 minutes at 100^0C. Thereafter the mould was allowed to bench cool to room temperature before opening. The specimens were inspected and

checked for voids, cracks, bubbles and for foreign objects incorporated in the PMMA.

4.2.4 Cyclic loading

Fatiguing of specimens is done by a process of cyclic loading where a repeated force is used to simulate use of the material in clinical conditions.

Fatigue, and the testing thereof by means of cyclic loading, is a specialized field in science. Due to the nature of dentistry, the specimens for testing are small and often fragile. This requires machines sensitive enough to produce, and reliably analyze relatively small forces.

Outside the Western Cape region, the CSIR in Pretoria had the necessary equipment and scientists able to operate the equipment. Also, the CSIR has a project to assist academic institutions in performing research projects and agreed to help with this study at a reasonable fee.

Vallittu (2006) describes 'fatigue strength' of a material as the highest stress that a material can withstand for 10^7 times. Testing specimens at such a high number of cycles poses a challenge in the laboratory milieu. The number of cycles per second must be kept low enough to prevent heat generation in the specimen. Thus, at 2 Hz, 57.8 days are required to fatigue one specimen for 10^7 times. However, in a review article, Naumann *et al.* (2009) found that a protocol using 10^4 cycles at 50 N and 5 Hz satisfactory simulated a year of function in dental materials. Cyclic load was thus applied for 10^4 cycles at 5 Hz. Each specimen was fatigued 10,000 cycles.

4.2.5 Flexural testing

Random specimens were selected from all 3 P/L ratio groups, both fatigued and non-fatigued. These pilot specimens were subjected to a 3 point bending test by the CSIR. All the specimens tested in all three P/L ratio groups displayed an adhesive bond failure between fibre and PMMA. Macroscopically it was noticed that a void surrounded the fibre bundle. This was an unexpected finding as the literature essentially stated the opposite (Bertassoni *et al.*, 2008). These results were in direct conflict with other studies and research papers read by the author except for a study done by Ladizesky *et al.* (1993) where they found that delamination may occur during some processing stages. However, the tests were conducted with highly drawn linear polyethylene (HDLPE) fibre, and not glassfibres as in my study.

The CSIR compared the mean FS of this pilot sample of specimens and there was no significant difference in different P/L ratio groups. The association with the failure pattern (Figure 3.1: Results), together with the lack of difference in FS results, were suggestive of the fact that the different mixtures of PMMA did not differ significantly in strength due to any interaction with the fibres. Of course as the fibres were lying in a void in the acrylic resin this was not surprising.

These unexpected preliminary results prompted an investigation into potential reasons for the adhesive failures encountered during piloting, as the aim of this study was to determine the influence of P/L ratio on the strength of the fibre re-inforcement of heat cured PMMA. This implied an efficient bond between fibre and matrix as pre-condition. At this stage it was suspected that the nature of the failure of the specimens was related to some step in the manufacturing process of the specimens.

These preliminary findings were communicated to the company who had read the research proposal prior to the study and then supplied the fibres.

CD's with images explaining every step of the process, my protocol and proposed methodology as well as a number of specimens of each P/L ratio were sent to the manufacturers in Finland.

Following suggestions from scientists from the manufacturing company, several issues were explored:

1. *The 2-stage method: The same PMMA but at different dough stages on each side of the fibre bundle were packed into the same cavity. Refer to communication with Pasi Alander - 6/2/2011 & 6/5/2011 (Addendum C3 and addendum C5).*

   Following this comment, specimens were made using the 2 stage technique, without fibre reinforcement. All the specimens were fractured using the 3 point bending test. There was no difference in the FS. No voids or air bubbles were noticed at the fracture interface or on the outside of the specimens where the 2 layers joined. This was suggestive that the PMMA at different dough stages was not the reason for the void formation along the fibre.

2. *The amount of monomer. Refer to communication with Pasi Alander - 5/13/2011 (Addendum C4).*

   The 3 groups with the different P/L ratios, including the group with the recommended ratio, had the same adhesive failure pattern.

3. *Partial polymerization of the matrix of the fibre bundle due to heat fluctuation during transport. Refer to communication with Pasi Alander - 6/5/2011 (Addendum C5).*

   A new batch of fibres in a special cooler box with controlled temperature was sent from Finland. New specimens were manufactured. A random selection of these specimens was subjected to the 3-point breaking test. Again there was a 100% adhesive bond failure between fibre and PMMA.

4. *Compatibility of the PMMA and the fibre. Refer to communication with Pasi Alander* - 7/12/2011*(Addendum C6).*

   Three different heat-polymerizing PMMA were used to manufacture the specimens. Again there was a 100% adhesive bond failure between fibre and the 3 PMMA's.This lead to the assumption that the presence of a fibre was instrumental in the formation of the void.

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5. *Polymerization cycle.*

   The polymerization cycle was modified as follows:

   a. Heat-polymerization by means of carefully controlling the temperature at 98°C, just below the boiling temperature of the water in the water bath. The rationale behind this is: should any gas develop during polymerization, that this would be limited to a minimum.

   b. Heat-polymerization at a lower temperature, but instead of the recommended 20 minutes, a conventional polymerization time of 6 hours was chosen. The rationale behind this was to establish whether the metal mould was maybe interfering with the heat transfer to the specimens and thus slowing down the proper polymerization.

   Changing the polymerization cycle did not influence failure pattern between fibre and the PMMA.

This piloting exercise consumed another batch of fibres. When the results of the piloting were communicated with the manufacturer, the manufacturer admitted that it was not known if the fibres used for this project were suitable to be used for heat-polymerizing PMMA. *Refer to communication with Pasi Alander 8/1/2011(Addendum C7)*

The manufacturer agreed to send different, non-preimpregnated fibres with a proven history of cohesive bonding between fibre and both cold- and heat-polymerizing PMMA. The handling of these fibres is different and more difficult compared to the impregnated fibres. *Refer to communication with Pasi Alander 8/31/2011(Addendum C2)*

The complete experiment was repeated using the batch of un-impregnated fibres. Regardless of the eventual outcome these specimens were to be accepted as the final specimens for testing.

4.3 Discussion of the results

The Stick Fibre is a unidirectional glass fibre bundle and should be used where high strength is needed for instance in full dentures or in composite bridge frames. (Figure 2.14: Methodology)

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According to instructions 'wetting' of the fibres with a slurry of sloppy PMMA is very important. This is not easy as the fibres separate when they are wetted and are then difficult to handle and position correctly. However, this was overcome and this type of fibre became the one used for the final methodology.

The fatigued and unfatigued groups each had a cohort of specimens of 100%, 90% and 80% P/L ratio and were all re-inforced with the un-impregnated fibres.

Fatiguing was done at the CSIR Laboratories, while the 3-point breaking tests were done at the Dental Faculty of the University of the Western Cape.

Analysis of results was done using only specimens that were intact (no bubbles, cracks, voids) and that did not display extraordinary readings (machine malfunction, computer glitches etc). This is the reason why the groups have slightly different numbers of specimens. Some specimens were also lost due to operator error while working the universal testing machine.

4.3.1 Macroscopic fracture patterns.

On examination it was found that the specimens with the un-impregnated fibres failed adhesively, the same failure pattern encountered as for the pre-impregnated fibres during the first piloting process. Every specimen fractured with the fibre debonding from the PMMA. Again it was found that the fibres lay in a void inside the heat cured PMMA. These voids appeared larger than the diameter of the fibre bundle.

As the aim of this study was to assess the influence of the fibre on the strength of heat cured PMMA with and without fatiguing, this observation would inevitably complicate answering the hypotheses.

However, an attempt was made to carefully examine the specimens and at least see whether certain trends could be observed among the different specimen groups.

4.3.2 Strength

Prior to testing, the trapezoidal cross-section of the specimens as they emerged from the mould, was machined and finished into a rectangular shape. This was done at the CSIR. A certain variation in width and height was noticed. The influence of this variation was examined and found to be a confounder (Figure 3.6: Results). The distribution of height width and length in the various mixratio/ fatigue subgroups was measured and plotted.
Notice that the 80% fatigued and unfatigued values do not even overlap.

Figure 3.7 (Results) shows boxplots of deflexion for the different ratio and fatigue subgroups. Within the fatigued = Yes and fatigued = No groups the trends, with the ratio are similar: mean deflex drops quite sharply from 80% to 90% groups and then does not change much from 90% to 100%. This is clear, i.e. the thinner mix bends more. Williams *et al.* (2001) also found that changing P/L ratio of four auto-polymerizing PMMA resins may have deleterious effects on the properties of the polymerised material: A lower P/L ratio resulted in significantly lower surface hardness and higher flexibility.

A boxplot (Figure 3.3: Results) of the subgroups using F max and ratio and fatiguing in the different subgroups shows a different trend. With specimen = No (unfatigued) the Fmax increases with the increased ratio, while with the specimen = Yes (fatigued) the trend is essentially not as clear. This is where the different confounders play a role.

To standardize these results a formula was used to find a covariate measurement for thicklr which could lead to an accurate calculation of actual strength of the specimens.

Flexural strength was used to standardize the measurements in all the three subgroups of groups 1 (Fatigued) and 0 (un-fatigued).

The results of the two groups (Table 3.21: Results) clearly demonstrate that the unexpected reversed trend illustrated in Figure 3.3 has now been corrected. In both fatigued and unfatigued specimens there is now a slight rise in strength from the 100% to the 80% mixture.

The fact that this study showed that the fibres do not actually adhere to the PMMA makes this result surprising. One would expect the specimens with the higher P/L ratio to be stronger. It can be postuated, however, that the more viscous 80% mix did in effect impregnate the fibres ever so slightly more than the stiffer 90% and 100% mixes of PMMA. This would explain the higher FS of the 80% mix in both the unfatigued and fatigued specimen groups.

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The increase in FS after cyclic loading, however, is an interesting trend to explore further in future studies. Could it be that cyclic loading results in an initial pseudo tempering of the acrylic?

4.3.3 Comparison of results with other studies

There are very few studies and research projects that concentrate on the fiber strengthening of heat-cured denture PMMA. Possibly this is because of the difficulty of using these fibres in PMMA that is, per definition, very thin and rarely exceeds 3.5 mm in thickness. In a study that compared heat-cure and microwave-cured PMMA fibre reinforced specimens Tacir *et al* (2006) found that strengthening with fibers lowered the flexural strength of the specimens but increased the flexural resistance. This compares favourably with this study. In this research study the manufacturer's instructions for all materials and fibres used was followed to the letter. The results are as published. In his 1999 study, Vallittu (1999) however placed great emphasis on the impregnating of the fibre bundles with monomer prior to use. It is possible that this change in the methodology allowed him to record the results he achieved.

4.4 Limitations and further research

In vitro studies have several limitations. The specimens are usually symmetric, unlike the variation and curvatures found in natural dentures. This is purposely done to control geometric variables and allow consistent loading on a flat surface in the same location for each specimen. The loading should also be consistent with other studies.

Clinical performance versus lab testing is a problem that has dogged researchers for a long time.

Clinical performance is classically defined in terms of safety and effectiveness. Dr. Gunnar Ryge, while in the employ of the United States Public Health Service (USPHS), came up with the most famous of the rating scales. This was considerably extended and the Modified USPHS Scale for Clinical Performance and Acceptability (Bayne, 2007) can now assess almost any dental procedure and material. Ryge isolated five variables or factors that he logically felt may describe many influences on clinical outcome. They include operator factors, design factors, material factors, intra-oral location factors and patient factors (Bayne, 2007).

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As an adjunct to this, exists the method known as ‘practice based research’ where the research is actually carried out in a real surgery environment of the dental practice. Of course here the clinicians’ different treatment decisions and their variations in assessment of clinical quality are a huge hurdle and a factor to be considered (Mjor, 2007).

It could be speculated that the relative difficulty and longer time it takes to fabricate the heat-polymerized specimens actually leads researchers to shun this group of materials in favour of the quicker and easier groups presented by the light- and auto-polymerizing resins.

The greatest limitation of this study is that the researchers could not achieve bond between the pre-impregnated C+B Stick fibres or the un-impregnated Stick fibres and the heat-cured PMMA. Up to this moment this result has not been explained either by this study or by the suppliers of the fibres in Finland. Not a single one of the ratio subgroups of either the un-fatigued or fatigued subgroups was significantly re-inforced by the addition of the fibres. This was clearly as a result of the debonding that took place between the heat-cured

PMMA and the Stickbond C+B and the Stick fibres. Despite numerous different approaches and techniques no method was found so far to successfully use Stick or Stickbond fibres with heat cure acrylic.

It could be argued that 'debonding' may be a misnomer as there may not have been a bond to start with. Jagger *et al.* (2003) found the same in their study with treated PMMA fibres where impact strength, modulus of rupture, modulus of elasticity, transverse strength and F Max were all negatively affected by the addition of fibres.

Once it was established that the bonding of the fibres was a problem, it could have been a good idea to use specimens with no fibres included as an additional control. This would have established with certainty whether the fibres bonded or not.

A further limitation of this study could be the 10,000 cycle load cap in the fatiguing process. In a previous study (Diaz-Arnold *et al.*, 2008) it was found that the 10,000 fatigue cycles had little or no effect on 5 different materials that were compared. The number and frequency of the cycles was based on previously reported literature, piloting and test time constrains. The testing time of every specimen at 10,000 cycles was 33 minutes (over 41 hours for all specimens). With the high demand for testing equipment, there is a tendency to limit cycling frequency.

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Within the two groups, 0 (un-fatigued) and 1 (fatigued), the wetter mix (80%) gave the highest FS, but the differences in the three subgroups were not significant. Therefore, the practitioner can change the P/L ratio to improve the handling for certain applications, without detrimental effect. Geerts and Du Rand (2009) also found no difference in FS for different ratios of un-reinforced chemically-cured PMMA. Since the PMMA used in my research did not bond to the fibres, the specimens could be regarded as 'un-reinforced".

All three the P/L ratios showed an increase in the FS after the fatiguing of the specimens from the 10,000 to the 20,000 cycle mark (Figure 4.1).This result was unexpected as the thought is that the cyclic loading (fatiguing) of the specimens would weaken them.

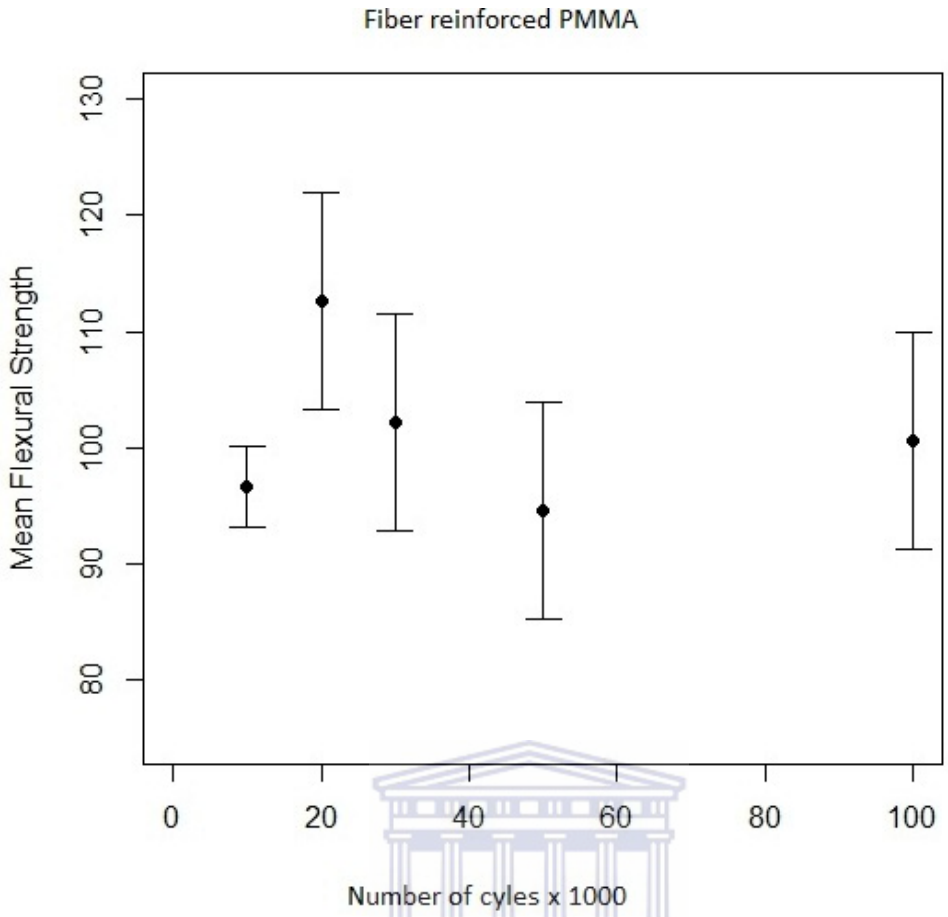

Figure 4.1: Plot of mean flexural strength against number of fatigue cycles showing an initial increase of strength with higher cycling

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The interesting phenomenon found by the CSIR that the specimens actually got stronger after cyclic loading (fatiguing) cannot be explained satisfactorily. One possible explanation could be that gentle cyclic loading actually anneals and aligns the PMMA chains in a similar fashion that tempering strengthens metals.

The fact that this result was achieved with heat-cured PMMA could also have a bearing on this result.

Further tests with a possible more vigorous loading cycle could be undertaken, to investigate whether this trend is short-lived and just takes place at a relatively low number of cycles.

Although the fibres in all subgroups were macroscopically fully debonded, Table 3.20 (Results) shows clearly the fact that: the wetter the mix, the stronger the specimen. This could possibly mean that even though the fibres do not successfully bond to the heat-cured

PMMA, the wetter mixes of the PMMA do have a slightly better adhesion between fibre and heat-cure acrylic. By examining fracture patterns, Geerts and Du Rand (2008) also found that adhesion between the wetter mixture of cold-cure PMMA and the fibre bundle was more efficient. In the case of chemically-cured acrylic, it did not result in a higher FS value though.

Tacir *et al* (2006) suggested that even pre-impregnated fibres should be soaked in monomer for 10 minutes to allow for better bonding with the acrylic resin.

The recommended ratio proposed for the PMMA used in this study proved to be the weakest mix of the three used. It could be possible that within the parameters of functional strength the manufacturers actually suggested the use of a mix that incorporated more powder in the fluid leading to increased consumption of the product.
Due to the fact that the specimens are manufactured and finished by hand, a certain variation in thickness and width was found. However, when variations were discovered, these variations were compensated for in the analysis and interpretation of the data.

4.5 Conclusions and clinical relevance

After exhaustive testing and using different PMMA materials and glass fibre bundles it was found that ever Stick and Stickbond glass fibre bundles do not bond to heat cured PMMA when using the recommended protocol and methods used in this study.

No reason for this could be established and exhaustive correspondence with the manufacturers of both the fibres and the PMMA has shed no further light on this problem.
In this study it was concluded that the fibre re-inforcing of heat-cured denture bases with this type of fibre is ineffective.

In vitro fatiguing results must always be interpreted with care. The assumption that the material with the highest FS after fatiguing would be the best or most appropriate material for the job at hand is not necessarily correct. Decisions on the selection for the most appropriate material should always be made within the broader clinical context.

The results of this study showed that, either debonding of the fibres and the heat-cured PMMA used in the study took place, or no bonding ever took place between the fibres and

PMMA.

Due to the cost of the fibres used for re-inforcement, it is imperative that the system should work as proposed. This cannot be achieved by using fibres in a heat-cure PMMA during flasking and processing of dentures. Possibly it would be better to use the method described in the Stick Company instruction CD and use a cold cure acrylic to insert the fibres after the denture in heat-cure acrylic has been manufactured.

Finally, it may be concluded that with regards to the PMMA:

1. There is no significant interaction between Mix and Fatigue state.
2. The 80 % mix has a significantly higher mean FS than either the 90% or 100 % mix (with differences of about 4.4 and 7.5 units respectively).
3. The Fatigued state has a higher FS mean than the Not Fatigued state (by about 6.0 units).

4.6 Recommendations

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The debonding of the fibres and the heat-cured PMMA or non- bonding between the fibres and PMMA was totally unexpected.
An additional study to examine other similar fibres from different manufacturers may help to identify a product that works optimally or to expose a flaw in the suggested use of these strengtheners.

FS (MPa) across all three P/L ratios increased after the fatiguing of the specimens from the 10,000 to the 20,000 cycle mark (Figure 4.1).This result was contrary to the widely held belief that the cyclic loading (fatiguing) of such materials would weaken them.
Research into this phenomenon could possibly lead us to be able to predict the behavior of acrylics used in dentistry more accurately.

The interesting phenomenon found by the CSIR that the specimens actually got stronger after initial cyclic loading (fatiguing) cannot be explained satisfactorily. One possible explanation could be that gentle cyclic loading actually anneals and aligns the PMMA chains in a similar fashion that tempering strengthens metals.
Research into this phenomenon could possibly lead us to be able to predict the behavior of acrylics used in dentistry more accurately.

The fact that this result was achieved with heat-cured PMMA and was not seen by Geerts and Du Randt (2009) in their research with self-cure acrylics could also have a bearing on this result.

Further tests with a possible more vigorous loading cycle could be undertaken, to investigate whether this trend is short-lived and just takes place at a relatively low number of cycles. Other different heat-cure PMMA materials could also be tested for comparison.

The recommended ratio proposed for the PMMA used in this study proved to be the weakest mix of the three used. Possible comparison with other dental acrylics would establish whether this was a single, product-specific finding or a definite characteristic of PMMA used for denture construction.

This researcher concluded that the fibre re-inforcing of heat-cured denture bases with this type of glass- fibre is ineffective.

No reason for this could be established and exhaustive correspondence with the manufacturers of the fibres and the PMMA used has shed no further light on this problem.

Correspondence with other manufacturers of glass fibre re-inforcing may shed light on this finding.

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Addendum C.2

From: Pasi Alander <pasi.alander@sticktech.com>
To: Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date: 6/5/2011 10:45 PM
Subject: VS: VS: comments about your samples

Dear Martin
some comments from me too. Now when you mentioned that fibres really dry, like matrix have gone, I started to think if the acrylic is dissolving the matrix away. This can happen if fibres are too long time inside unpolymerized acryl. But what makes empty space around them. I don't know.
I was also wondering if the two stages of the acrylic will do the porosities in to the acrylic. This porous might be as a one big empty area around the fibre. Does the two stage acrylic technique affect to the acrylic strength values, I don't know. This can be tested by fabricating some control samples with two different method. In a first group the mould is filled once with acrylic and in a other group with two step technique.
Also the reason can be that the heat can polymerize the fibres during the transportation. everStickC&B fibres should be totally flexible when using those. There should be also thin oxygen inhibition layer around the fibre bundle after light polymerization. Don't use vacuum or place fibres inside the silicone while polymerizing those with light. We can send new fibres for you with the data clocker. It will tract the temperature of the parcel from here to you.
Your test sample size is so big that you should put more fibres in to the test samples. Now the fibre amount by volume is less than 2 %. This is not enough for getting proper reinforcement effect. You will need 2-3 bundles at least in one test sample to find out differences between the control group without fibres and reinforced group. Is that possible? Samples can also be smaller if possible. As I probably told you earlier, by adding the stick fibres also to this study it will be more informative. But maybe too much work with this little time.
We can send the fibres directly for you by TNT, it needs your address and phone number.
Best regards
Pasi
Pasi Alander
Product Manager

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Lähettäjä: Martin Stuhlinger [mailto:mstuhlinger@uwc.ac.za]
Lähetetty: 3. kesäkuuta 2011 14:52
Vastaanottaja: Pasi Alander
Aihe: Re: VS: comments about your samples

Addendum C.3

From:	Pasi Alander <pasi.alander@sticktech.com>
To:	Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date:	8/1/2011 2:46 PM
Subject:	VS: my newest results.
Attachments:	5 1018 Stick product family updated 2011_04 low res.pdf

Dear Martin
I just came back from summer vacation. I do not have clear answer to this problem. Reason can be that everStick fibres are not working well with heat cured acrylic.
The acrylics we have used with everStickC&B fibres are mostly self-cured acrylics, like Palapress from Heraeus Kulzer. We do have that much experience with heat cured acrylic products. Test with everStickC&B and heat cured acrylics have not been done, because the other our fibre, named Stick, is fully tested. These tests showed that it will work well with both types of acrylics (self and heat cured). That makes us believe the same with everStickC&B. You have proved that we were wrong. everStickC&B fibre can be used with self-cured acrylic, but is maybe not suitable for heat cured acrylics. It is very valuable information for us.
We do still have reinforcing product for heat cures acrylic. it 's name is Stick. See the different fibre products dental laboratories in the attachment.
Best Regards
Pasi

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Addendum C.4

From: Pasi Alander <pasi.alander@sticktech.com>
To: Martin Stuhlinger <mstuhlinger@uwc.ac.za>
Date: 8/31/2011 12:33 PM
Subject: VS: VS: VS: my newest results.

Dear Martin
Nice to hear that can continue your study.
I want highlight one more time that Stick fibre need wetting with slurry acrylic mixture before placement. I hope this is not problem for the research question/topic. Use metal instrument for manipulating fibres during the wetting to ensure the proper wetting.
Both everStickC&B and Stick have 4000 single fibres in one bundle. But Stick fibre needs more hand skills than everStickC&B. After wetting all 4000 single fibres are loose from each other, because wetting acrylic will dissolve totally the porous PMMA matrix of Stick fibre. Fibre bundle will swell also some amount. The final diameter will be more than 1.5mm. This makes the handling little bit tricky. Use two tweezers (both ends) to lift the fibres in to the right position. I just want to inform you beforehand these things, which might affect for sample fabrication.
I will start my on study at October. I will be totally away from work more than one year.
Please contact to the Eija Säilynoja if more information is needed.
You will get the e-mail when we sent the fibres to you.
Best Regards
Pasi

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Product Manager

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