6 New cyclic loading technique for measuring fracture toughness of paper

6.1 Introduction

As has been established in Chapters 1, 2, 4 and 5 there is a need for a technique that can rapidly assess the fracture toughness of paper. Since paper is a visco-elastic material, it shows significant plastic deformation under stress. The drawbacks in the non-linear fracture mechanics methods, such as the Essential Work of Fracture (EWF), that was developed to estimate the fracture toughness of ductile materials have been discussed in previous chapters. The major difficulty in applying the EWF technique to paper is that it requires testing a large number of samples of different sizes. The EWF technique has never been considered to be a quality control tool in paper manufacturing or converting, since the required time for a single measurement is far greater than the time taken for a typical quality control testing cycle. In addition the significantly large sample area required for the EWF technique is another serious disadvantage, especially when measurements are carried out on laboratory made sheets. The laboratory manufacture of handsheets consumes significant time and labour and a technique that can reduce the sample area required for measurements is highly desirable.

6.2 Alternative approaches of developing a new technique

To develop a new technique for measuring FT of paper a number of methods were examined. The speed, accuracy and the requirement for a measurement at only one sample size were the important features targeted from a new technique. One approach was to select a fairly small sample dimension prepared to deep double edge notched tension (DENT), to give an approximate ligament length of 2mm. The reason for selecting a small sample was to reduce the amount of plastic work consumed during a fracture, as it was assumed that the plastic work W_p scales with the square of the ligament length in a DENT sample (Cotterell and Reddel 1977). One of the essential criteria that have to be met in the measurement of in plane FT is that the ligament of the sample should be under an in-plane stress. However when the ligament is small, it can only partially satisfy this condition. Hence the method was not entirely successful.

Another approach was to load and reload the sample around its average breaking loads to estimate the work consumed for the plastic deformation. This method required two sets of samples where one set was initially loaded for complete failure to obtain the average failure load for a given sample dimension. The second set was then loaded and unloaded when the sample reached the average breaking load. The area in the cyclic load-extension curve was assumed to be equal to the work consumed for plastic deformation. This plastic work was then removed from the total area obtained from the full load-extension curve, with the remaining work giving the fracture toughness of the sheet. This procedure was not very successful for a non-uniform material like paper for the following reasons. Those test pieces that failed at loads lower than the average were completely rejected and hence more pieces were required to replace the rejected ones to obtain a reasonable average. The test pieces that reached the average failure load (maximum load before reloading) were obviously failing at higher loads than the average and hence these test pieces were not representative of the whole sample. This could produce an inaccurate estimate of the non-essential plastic work of fracture. It also appeared that the fracture toughness measured with this method was dependent on the sample dimensions.

In another approach, a set of 4 or 5 segments or perforations were introduced in the ligament area to minimise the plastic deformation during fracture. This method showed some promising results for brittle samples but underestimated the fracture toughness of tough ductile samples. The most promising approach was found to be the cyclic-loading method and details of this technique are discussed in the next section.

6.3 Cyclic loading technique for the measurement of sheet fracture toughness

6.3.1 Test Method

The technique developed in this work was to cyclically load a sample cut in DENT geometry, increasing the maximum load in each cycle. It is believed that when a paper sheet stretches, some inter-fibre bonds open progressively. The elongations of the stretched fibre segments are partially irreversible. This loading and unloading process consumes energy and only part of that work is recoverable in elastic recoil from unloading with the rest consumed in irreversible deformation. This characteristic of paper was utilised to "cycle out" the work consumed for the plastic deformation and effectively separate the essential work consumed in the fracture process zone from the total work. The pre-conditions for a successful test with the cyclic technique are the same as those for the EWF technique, so that the upper limit of the ligament length is determined by the size of the plastic region and should satisfy the condition $L_{upper} \leq 1/3B$, where B is the sample width. The lower limit of the ligament is governed by the sheet thickness and is of the order $L_{lower} \geq 5t$. The other pre-conditions for the technique are that the ligament completely yields before crack initiation and that the sample is strained under plane-stress.

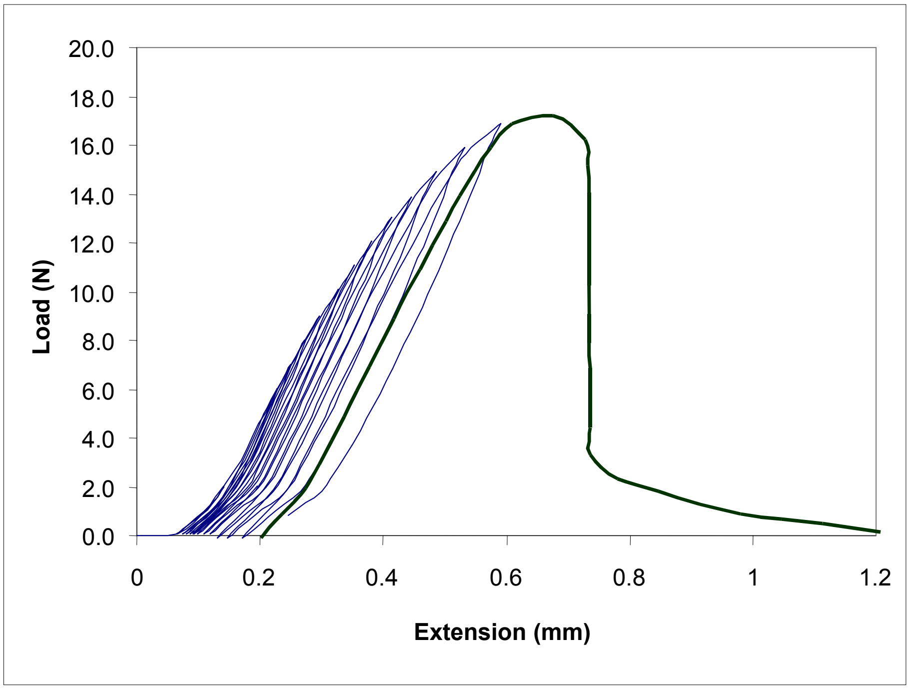


Figure 6.1 Cyclic load-extension curve of a DENT sample (L=5.1 mm, span=90 mm) cut from ultra-low coarseness radiata pine refined for 15 minutes

A typical measurement carried out from a sample made from ultra-low coarseness radiata pine fibres refined for 15 minutes in a Valley beater, with a ligament of 5.1 mm, is shown in Figure 6.1. The last cycle, where crack propagation begins and fracture is completed, is marked with a thicker line in the figure. As can be seen from the figure, the elastic modulus in the final cycle before the sample fractures is approximately the same as that in the initial cycles, although the sample showed significant plastic deformation of 0.2 mm. As the modulus of the final cycle is approximately the same as the modulus of initial cycle, we can assume that no significant fatigue damage has occurred during the cycling process.

The loading cycles themselves were conducted under a mixture of displacement and load control. Initially the sample was loaded at a rate of 2 mm/min, until it reached a set maximum load, after which it was then unloaded at a rate of 10 mm/min. The unloading rate reduces the time required for each measurement. Normally the sample was unloaded only until it reached a small positive load (approximately 1 N), to avoid any damage to the ligament area from bending. In the measurements shown in Figure 6.1, the set maximum load has been incremented by 1N after each cycle. During the final cycle the specified load was never reached since the sample begins to fracture. At the same time the software that controls the loading continuously increased the displacement at a rate of 2mm/min until the sample completely fractured. The test was then manually terminated when the sample had completely fractured. If the fracture had started before the final cycle, this can be clearly identified by the cyclic load-displacement curve. That is, the maximum load achievable in the last cycle will be reduced compared to the previous cycle. This reduction is an indication of the reduction in the ligament length due to the propagation of the crack.

The work consumed in plastic work in all of the cycles prior to the final cycle is not essential to the work of fracture and this work must be associated with the work done in the outer plastic zone. The work under the final cycle is the only work, which is essential to the fracturing the material, and is expected to be equal to the EWF fracture toughness. The method of “cycling out” the non-essential work from the essential fracture work provides an easy way of separating the two without testing the samples at multiple ligament lengths.

There are many potential advantages of this cyclic technique over the EWF technique. One main advantage is that the cyclic technique requires only one sample size. This greatly reduces the number of tests generally required to measure FT, in comparison to the EWF technique. Further the selection of one, relatively small ligament length significantly reduces the sample area required for testing. For a typical test sequence, with a ligament length of 5 mm, the cyclic test uses around 10% of the sample area consumed in the EWF test. When compared with J-integral fracture toughness measurements, as implemented in the Lorentzen and Wettre (L&W) instrument, the cyclic technique requires only 20% of the sample area used in the J-integral technique.

The cyclic method can also be more readily automated than the EWF technique, as all the tests can be conducted at one ligament length. In this work we used the dimensions of L =5.1 mm and 90 mm span for the dimensions of the DENT samples. This overcomes the difficulties in automating the sample cutting and testing for the EWF technique. The cyclic method can also be further automated to directly estimate the fracture toughness, since the fracture toughness can be calculated from the area under the final loading cycle. The technique shows a great potential to expand the useability of the EWF technique. One disadvantage of the cyclic technique is that each cyclic test takes longer than a normal EWF test carried out on a single ligament due to the cyclic process itself. The strain rate maintained in both the cyclic and EWF tests was 2mm/minute.

The time taken to perform a single cyclic test was minimised by using an unloading strain rate was of 10mm/minute. The testing time was further reduced by setting the maximum load in the first cycle to 4N below the average maximum load, which minimised the number of cycles required before fracture occurred. It was found that the measured results do not depend on the number of cycles prior to fracture. Ideally, the maximum number of cycles required for a measurement is only two, where one cycle is the loading/unloading cycle to a point just before the fracture is initiated after which a final loading cycle completely fractures the sample. However, this procedure is not suitable for a non-uniform material like paper, because it is impossible to estimate the load at which a given sample breaks with sufficient accuracy. The application of a two cycle procedure may be successful for a more uniform material with a well-characterised breaking load.

The comparison of data obtained from the EWF and cyclic techniques for the ultra-low coarseness sample is shown in Figure 6.2. The figure shows the full EWF plot

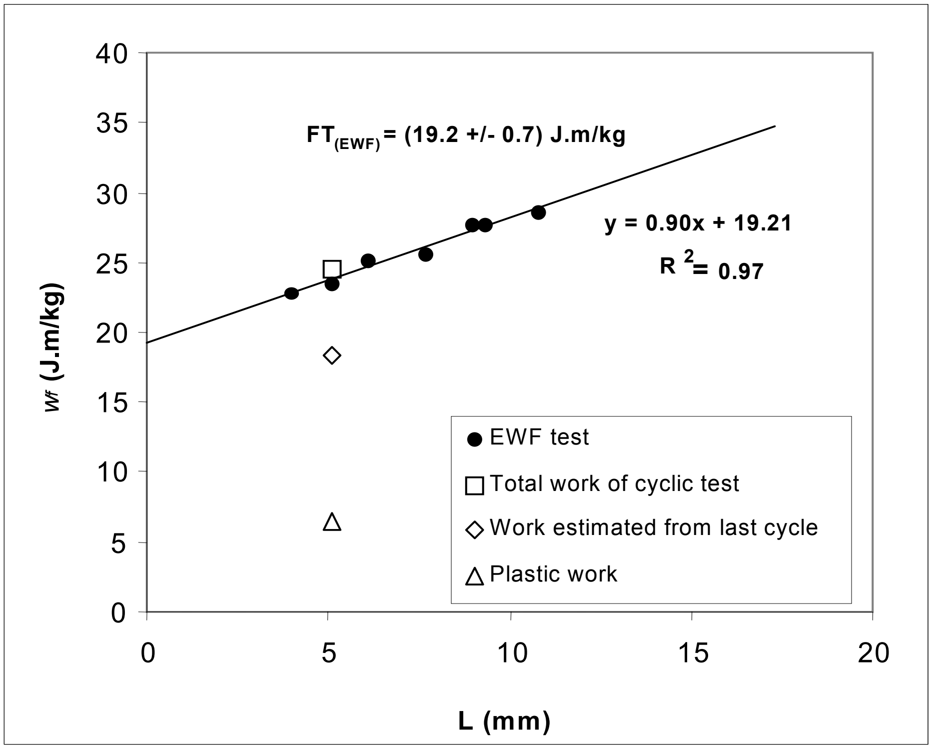


Figure 6.2 Fracture toughness estimated from the EWF technique and by the cyclic loading technique for sheets made from ultra-low coarseness radiata pine pulp refined for 15 minutes

where w_f (= $W_f/L.G$) is plotted against the ligament length (L) of the DENT sample. Here G is the grammage, which was 63 g/m^2. Each data point was the average of 15 tests. The linear fit to the EWF data gives the y-axis intercept of 19.2 ± 0.7 J.m/kg. The filled circles shown in the figure are the data points obtained from the EWF measurements while the open points are obtained from the cyclic measurements. The three open points are (i) the work measured in the last cycle (after normalising by ligament length, L, and the sample grammage G) (diamond) (ii) the total work estimated from cyclic, load-elongation curve (square) and (iii) non-essential plastic work (triangle), which is estimated as the difference between the total work and the work in the last cycle.

The work of fracture estimated from the cyclic-loading method was 18.2 ± 0.9 Jm/kg, which is 1.0 Jm/kg less than the value determined from the EWF method. However these values are equal within experimental errors. Another important outcome observed in these tests is that the total work measured in the cyclic method is the same, within the errors, as that measured in the EWF method. This is very important evidence against the repeated cycling having caused any fatigue damage. If fatigue damage has occurred, then additional fibre fracture and fibre matrix de-bonding will occur around the crack tip. Then the specimen after cycling would be different to that of an identical specimen that had been monotonically loaded to the same maximum load. The fact that the total work done on the fracture process has remained the same, whether the samples had been fractured in cyclic loading or monotonic loading indicates that there has been no fatigue damage.

6.3.2 Test conditions

A successful test with the cyclic technique should satisfy same pre-conditions as those for the EWF technique. These conditions are; (i) the ligament (L) should satisfy the condition, $5t \leq L \leq 3B$ and remain in plane stress and (ii) the area around ligament should yield completely before the sample fractures. The literature also suggests (Seth, Robertson et al. 1993) that the crack propagation should be stable (no brittle fracture) in order to successfully measure the EWF fracture toughness.

There is no straightforward theoretical method of determining the limiting length of a sample for stable fracture. This is because the paper mechanical properties such as strength, stiffness etc., that are required for such an estimation, do not directly correlate with fracture toughness (Wanigaratne, Batchelor et al. 2002), (Yu and Karenlampi 1997). In this work the effect of sample length on cyclic fracture toughness was investigated using a tough material made from high coarseness radiata pine refined for 75 minutes. In this investigation DENT sample length was varied from 90 mm to 25 mm and ligament was kept to a constant, at L=5.1mm. The DENT samples of ligament length of 5.1mm and length of 90mm were the standard sample size used for all the other cyclic tests. Two representative cyclic load-elongation curves for sample lengths of 90 mm and 25 mm are shown in Figure 6.3. It is evident from the figure that the curve for the 25mm long sample shows continuous stable fracture, while a part of the unloading curve for the 90mm long sample shows unstable fracture. It is further evident from the figure that the stored elastic energy for the 90mm long sample was insufficient to complete the fracture as the brittle failure stops and a long tail occurs in the failure curve extending about a millimetre further. The reason for the long tail was due to unbroken fibres bridging the fracture surfaces even after the crack has propagated past the fibres. In order to achieve complete fracture these fibres must be pulled out against the bonds that hold them.

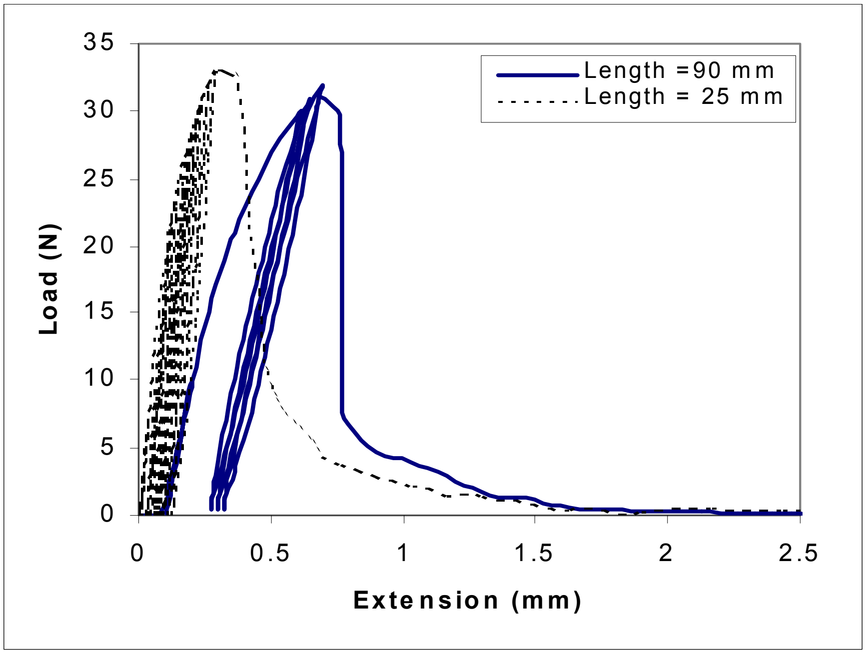


Figure 6.3 Cyclic load-extension curves from 25 and 90 mm span specimens for high coarseness radiata pine refined for 75 minutes

Figure 6.4 shows the measured cyclic fracture toughness as a function of sample length. It is clear from the figure that the measured fracture toughness is independent of the sample length of this material. This suggests that if either fracture is stable or partially unstable (for 90mm sample length), then the cyclic fracture toughness is independent of sample length. This can be further rationalised in the following way.

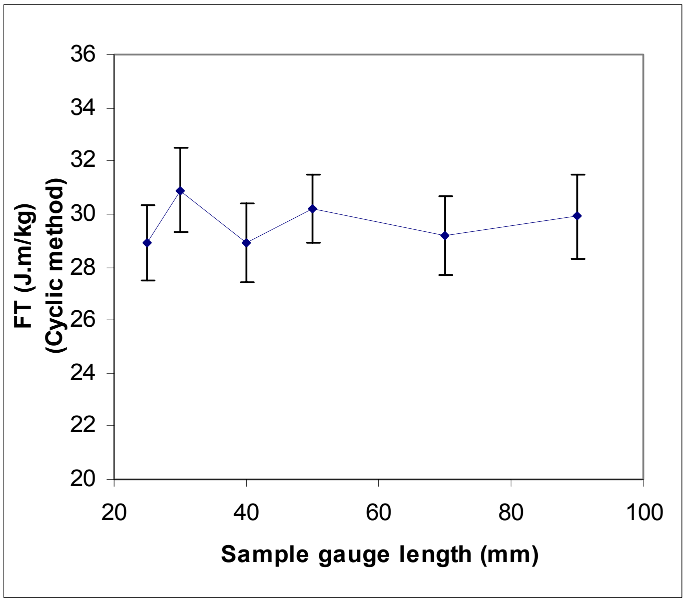


Figure 6.4 Cyclic fracture toughness measured at different sample gauge lengths for highly refined (75 minutes) high coarseness radiata pine samples with ligament lengths of *L*=5.1mm

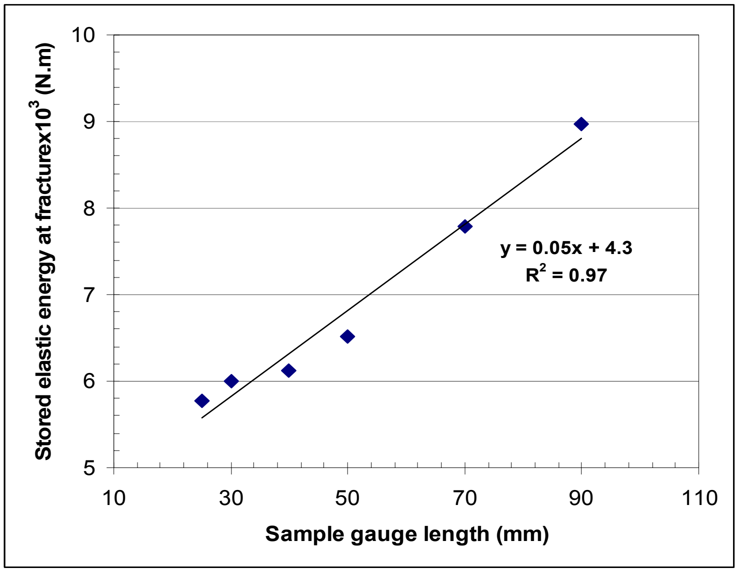


Figure 6.5 The variation of stored elastic energy at fracture with sample gauge length for L=5.1 mm DENT samples

The stored elastic energy of a sample with constant ligament length increases with increasing sample length and the breaking force. Figure 6.5 shows the variation of stored elastic energy as a function of sample gauge length, L=5.1mm, DENT samples.

If the stored elastic energy is just sufficient to complete the fracture then the energy absorbed in crack propagation is equal to the fracture toughness. However, if the stored elastic energy is greater than the required energy to fracture the sample, the measured energy in the last cycle would be the stored elastic energy, which would be an overestimation of the fracture toughness. Although the 90mm long sample has failed in a partially unstable manner, where the stored elastic energy was enough to propagate the crack through the ligament, there was not enough energy to separate two crack faces by pulling out all the fibres that bridged between the two crack faces. This process can be labelled as *partial* brittle fracture, in contrast to *complete* brittle fracture, in which the stored elastic energy is sufficient to fully fracture the material. It has previously been assumed that the fracture process must be completely stable in order to accurately measure fracture toughness with the EWF technique (Cotterell and Reddel 1977; Seth, Robertson et al. 1993). However, the results showed in Figure 6.4 contradict this assumption and show that the EWF and cyclic fracture toughness can also be accurately measured even if partial brittle failure occurs.

6.4 Comparison of essential fracture work estimated from EWF and cyclic-loading techniques

A comparison of fracture toughness measured from EWF and cyclic techniques for 39 commercial and laboratory made papers is shown in Figure 6.6. These 39 samples consisted of 8 machine made samples, described in Section 3.3 and 31 laboratory made handsheets. A summary of the samples details and the results is given in Appendix C. The laboratory made sheets included 12 from previously dried, bleached New Zealand radiata pine pulps (high, medium & ultra low coarseness) and 19 from never-dried unbleached Australian pulps. Details of these pulps and the preparation of the handsheets were given in Section 3.2. Handsheets were made from unrefined pulps and pulps that had been refined in a Valley beater for up to 75 minutes using a Moving Belt Sheet Former (see details in Chapter 3). All the commercial papers were tested in both machine (MD) and cross directions (CD). Newsprint samples were tested only in MD direction. The results of some of the extended testing carried out on machine made papers are given in Appendix D.

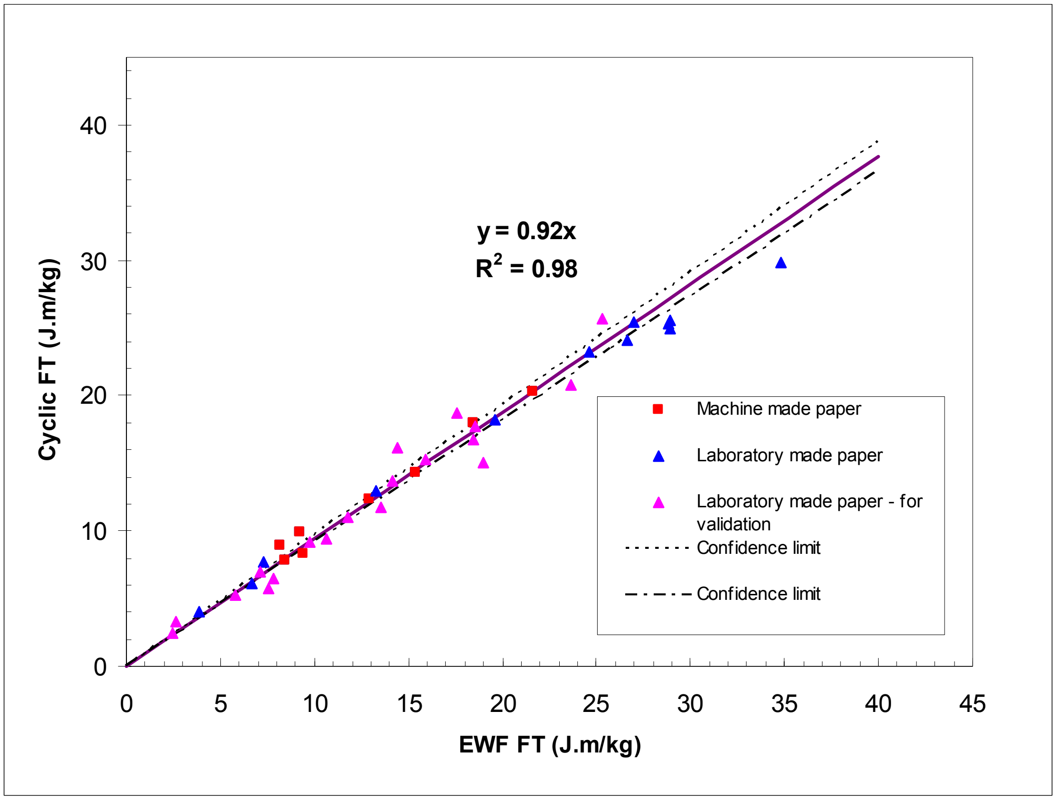


Figure 6.6 Comparison of EWF and cyclic fracture toughness data for 39 test specimens

The fracture toughness values estimated from EWF technique range from 4 to 35 Jm/kg. It can be seen from the Figure 6.6 that the fracture toughness values estimated from the cyclic loading technique are on average 8% lower than the values estimated from the EWF technique. Only a limited range of machine made samples were tested during the course of the work presented here. Subsequently, other workers have compared cyclic and EWF fracture toughness for machine-made papers manufactured by SCA in Sweden (Kelly 2002; He, Batchelor et al. 2004) and newsprints manufactured by Norske Skog (Felicia and Hambali 2004) .

The straight line fit to the data set in Figure 6.6 gives a slope of 0.92 ± 0.01, with R^2 = 0.98. This fitting was obtained after including the estimated errors in the determination of each data point and forcing the line through the origin. The SCA made papers included test liner, kraft liner, supercalendered paper (SC) and light weight coating (LWC) basestock. These data are shown in Figure D1 in Appendix D1, together with two data points for polypropylene films (Kelly 2002). The results shown in Figure D1 are in complete agreement with the data shown in Figure 6.6 as a linear fit and gives a relation of y = 0.93x with R^2 of 0.94.

Since a small amount of work from plastic deformation could be present in the final cycle, it would have been expected that a higher fracture toughness would have been obtained from the cyclic method compared to the EWF method. However, the results proved otherwise. The proportion of work from the outer plastic zone included in the final cycle will depend on the size of the increment in the maximum load in the successive loading cycles and this increment was kept as small as possible (1N). With the aim of further investigating the reasons for lower values of cyclic fracture toughness over the EWF method, the cyclic fracture toughness of three samples was measured over a range of ligament lengths and compared to the results with the fracture toughness measured from EWF method.

6.5 The effect of ligament length on cyclic fracture toughness

It is also important in obtaining cyclic fracture toughness at a range of ligament lengths to examine any effect from ligament length on cyclic fracture toughness. This also helps to identify any dependence of cyclic fracture toughness on sample dimensions or geometry. The paper samples used in the measurements were laboratory hand sheets made from high coarseness radiata pine refined for 75 minutes and commercial plaster liner-board**,** tested in MD and CD directions. One advantage of the cyclic technique over the EWF method is that the fracture toughness can be obtained from samples having different ligament length, so that the effect of the sample geometry on the results can be tested.

Figure 6.7 shows the data for the highest toughness material, the high coarseness radiata pine refined for 75 minutes. It can be seen from the figure that the fracture toughness of this strong, tough material is independent of the ligament length, for the range of ligaments tested (4.0 to 14.1 mm). The fracture toughness estimated from the EWF technique was 34.6 Jm/kg, while the average fracture toughness measured from the cyclic technique was 29.4 Jm/kg. This difference cannot be due to some plastic work in the outer plastic zone being included in the final cycle. As if that were the case then it would produce a cyclic fracture toughness which is higher than that estimated from EWF method. This difference can only be explained if it is possible to identify some work performed on the sample that scales with the ligament length, but occurs before the fracture.

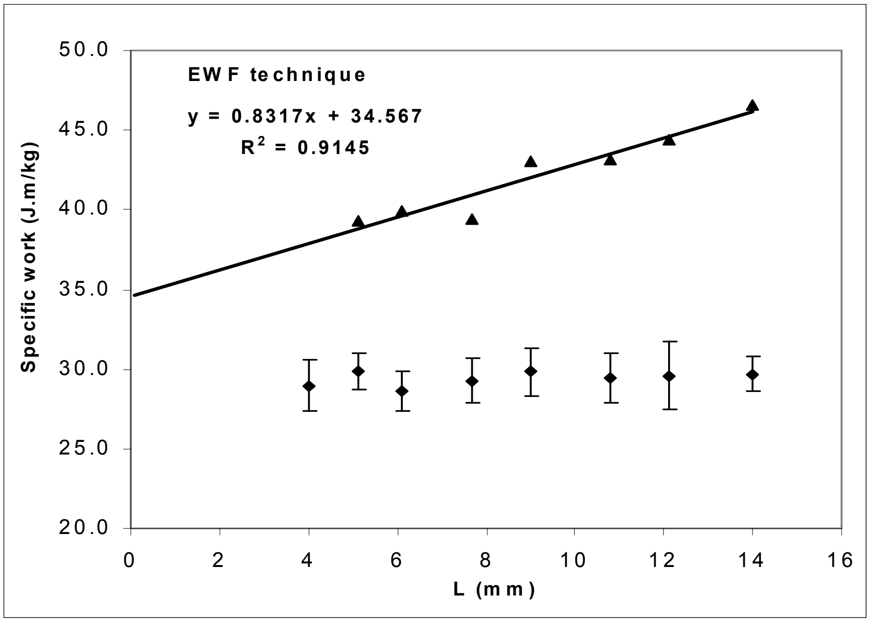


Figure 6.7 Comparison of the fracture toughness measured from EWF and cyclic technique at different ligament lengths for high coarseness radiata pine refined for 75 minutes

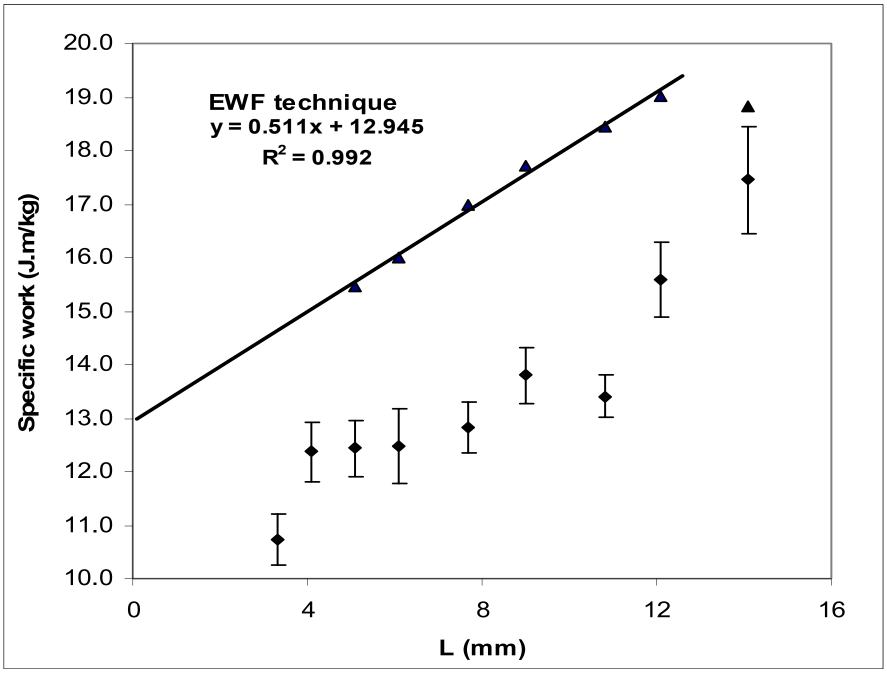


Figure 6.8 Comparison of the fracture toughness measured from EWF and cyclic technique for plaster liner-board measured in the machine direction (MD)

Figure 6.8 shows the data obtained from the tests on plaster liner-board tested in MD direction. The cyclic fracture toughness values were estimated for samples with ligament lengths varying from 3.3 to 14.1 mm, while the EWF tests were done on samples with ligament lengths from 5.1 to 14.1 mm. The data points fitted with a straight line shows the EWF test results. The fracture toughness measured from the EWF fit was 12.9 ± 0.7 Jm/kg. The EWF points were fitted well with a straight line except for the point at the largest ligament, 14.1 mm. The data at this length are consistent with a ligament which hasn't completely yielded prior to the crack propagation, producing a type ii or iii stress field at a failure, and thus an incorrect result (Tanaka, Otsuka et al. 1997). It is necessary that a DENT sample should display a type i (a circular or elliptical deformation field) before the sample fractures, to satisfy the condition for a successful EWF test.

The results presented in Figure 6.8 show that the cyclic fracture toughness values are consistent within their uncertainties for ligament lengths from 4.0 mm to 10.8 mm. The cyclic fracture toughness estimated for the samples with ligament lengths of 4.0, 5.1 and 6.1 mm was 12.4 ± 0.6 J m/kg while the highest ligament lengths of 12.1 mm and 14.1 mm had cyclic fracture toughness of 15.6 and 17.3 Jm/kg, respectively. It is interesting to observe that the value of cyclic fracture toughness for 14.1 mm ligament length is close to the total energy measured at the same ligament length in the EWF test, which was 18.7 Jm/kg. This indicates that a large amount of plastic deformation has occurred during crack propagation, and that most of the work in the outer plastic zone has occurred after the crack started to propagate. The large amount of plastic work occurring after the crack has begun to propagate has produced errors in both the EWF and the cyclic measurements at this ligament length.

The comparisons made between EWF measurements and cyclic fracture toughness measurements at different ligament lengths for CD plaster liner-board are shown in Figure 6.9. The fracture toughness estimated from EWF technique gives a value of 9.3 ± 0.3 Jm/kg, which is lowest among the three tested samples. The results presented here from cyclic fracture toughness technique show a weak but gradually increasing trend with increasing ligament length, for ligament lengths between 3.3 and 9.0 mm. For larger ligament lengths, the measured cyclic fracture toughness is constant and equal,

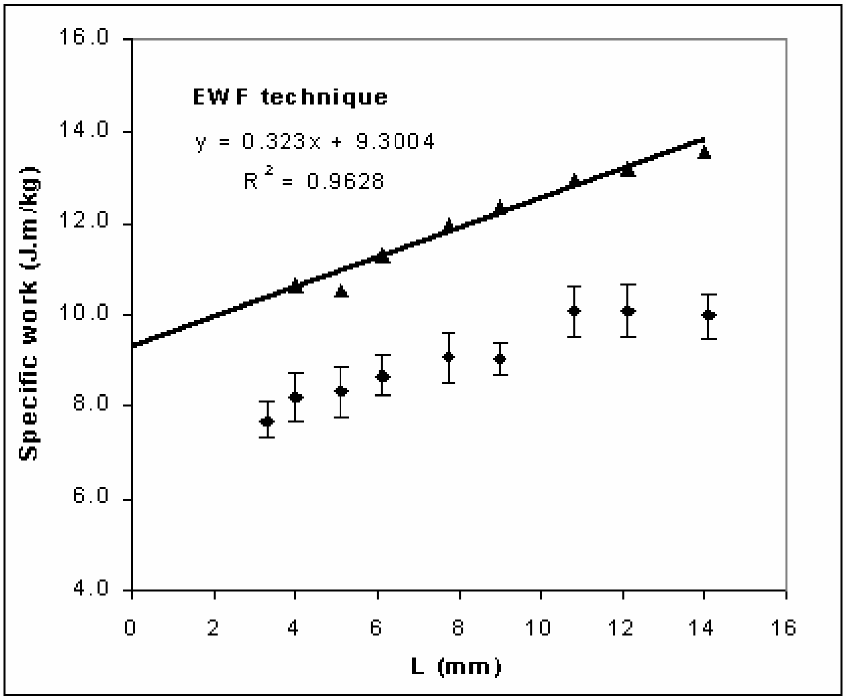


Figure 6.9 Comparison of the fracture toughness measured from EWF and cyclic technique at different ligament lengths for plaster liner-board measured in cross-machine direction (CD)

within errors, to the EWF fracture toughness. It was found that the tensile strength of the samples tested in the CD direction was only 40% of the strength in the MD direction so it was not until the ligament length was greater than 9.0 mm that the force required to fracture the sample was equal or greater than the force required to fracture the MD sample with the 4.0 mm ligament length.

Subsequent to the work in this thesis the measurements in Figures 6.7-6.9 were repeated for “Reflex” copy paper in MD direction (Sudarno and Nicholson 2003). The results are shown in Appendix D2. They measured the cyclic fracture toughness for ligament lengths in the range 3.3 to 9.1 mm and cyclic fracture toughness was approximately constant across this range with a measured average value of 7.7 J.m/kg, compared with a value of 9.9 J.m/kg determined for the EWF fracture toughness.

6.6 Discussion

The results shown so far suggested that there is a great potential to use cyclic fracture toughness technique in the industry to determine fracture toughness as a QC measurement.

To establish this technique as a standard, an explanation for the difference between the cyclic and EWF fracture toughness of all test materials shown in Figure 6.6 is necessary. In order to explain this discrepancy, a deformation work that, scales with ligament and occurs prior to the fracture, will be required. This work should also not be part of the plastic work consumed in the elliptical or circular deformation field that is developed before fracture (Wiens and Gottsching 1999; Tanaka and Yamauchi 2000; Wanigaratne, Batchelor et al. 2000). This is because the work consumed in the outer plastic zone will scale with the square of the ligament length of the sample, provided the sample has a type i plastic deformation field.

Kettunen et al (Kettunen and Niskanen 2000; Kettunen, Yu et al. 2000) have shown that microscopic damage zones are formed around the crack tips before samples begin to fracture. These are formed by bond and fibre fracture around the crack tip and continuously grow as the load is increased to the failure load. These zones can extend up to 6 mm. More importantly the work required to form these damage zones will not be included in the work in the last cycle.

It is difficult to prove that the work in forming these damage zones around the crack tips is the same as the difference between the EWF and Cyclic fracture toughness. This is because the work required to produce these zones has never been estimated. However, for a given sample, the Crack Tip Opening Displacement (CTOD) will determine the extent of the damage zone around the crack tip, because the CTOD determines the local strain at the crack tip.

Mai and Cotterell (1986) showed that for EWF testing of ductile polymeric materials the total measured work can be divided into a work of fracture initiation, W_i and a work of fracture W_f. The W_i is the work to create the necked zone that precedes the fracture of the sample. When the CTOD at failure is independent of ligament length, the degree of necking is approximately proportional to the ligament length. Mai and Cotterell showed that for the polymers they tested $w_i = W_i/Lt$ is approximately constant. They argued that if the work required to create a given CTOD is proportional to the ligament length, the w_i will be a constant and this was confirmed in the measurements.

This is likely to be true for paper as well. For a given ligament length the work required to produce a given CTOD and thus a damage zone will be proportional to the ligament length of the sample. The work of fracture initiation or the work required to create damage zones will be a constant and is possibly equal to the difference between the cyclic and EWF work of fracture toughness. This view correlates well with the comparison data presented in Figure 6.6, which shows the cyclic fracture toughness is around 8% lower than the corresponding EWF toughness, independent of the measured value.

Tanaka, Yamauchi (1999) measured the CTOD of paper using a method of printing fine dots and analysing the movements of the dots as the sample is strained. They reported that the critical CTOD ($CTOD_c$) of a machine made sack kraft in both MD and CD was constant for ligaments greater than 9mm. The $CTOD_c$ was lower for ligament lengths of 5 and 3 mm. This material falls into the same general class of materials as the plaster liner boards, for which results were presented in Figures 6.8 and 6.9. A possible cause for the reduction in the $CTOD_c$ at the small ligament lengths could be because the ligaments are small enough so that damage zones, formed around the crack tips, begin to interact with each other. However, this can not be definitively proven until the damage zone sizes of the samples are tested, which is something that was beyond the scope of this thesis. This interaction of damage zones is also a possible cause of the lower cyclic fracture toughness values observed in Figure 6.8 and 6.9 for smaller ligament lengths. In these Figures, a reduced value is shown for ligament lengths less than 4 mm for the MD samples and less than 11 mm for CD samples.

If this hypothesis were correct, then sheets made from refined pulps should show smaller damage widths than the unrefined pulps. The refining process acts to improve the bonding between the fibres and the sheets showing high fracture toughness are the ones that pulps that have been heavily refined. Kettunen *et al* reported the typical values for the damage width of sheets made from heavily refined softwood kraft, and these were only 1-2 mm. The reason for having constant cyclic fracture toughness values, for all the ligament lengths tested for the heavily refined high coarseness radiata pine, is that the damage zones may be small enough so that even for a 4mm ligament they did not interfere with crack length.

6.7 Prediction of paper failure strength using fracture toughness

Niskanen *et al* (2001) proposed the use of a modified equation derived from linear elastic fracture mechanics (LEFM) to predict the tensile strength and apparent tensile strength of paper. The equation used was;

$$\sigma_{app} = \frac{\sqrt{G_c E}}{\beta\sqrt{2\pi(a + w_d)}} \qquad \textbf{(6.1)}$$

Here G_c is the fracture energy and E is elastic modulus of the material. The geometric factor β depends on the defect size, a, and the width of the specimen. One of the significant features in this equation is the inclusion of the damage width (w_d) of the fracture, in place of the theoretical correction term in Irwin's model (Irwin 1957). Niskanen *et al* (2001) described the damage width as the area in which bond failures and other microscopic fractures occur. They have investigated the change in damage width with fibre properties such as fibre length, strength, inter-fibre bonding and fibre segment activation. They reported that when fibres were made shorter, the damage width decreased with mean fibre length and the damaged width was approximately equal to the length weighted fibre length (Kettunen, Yu et al. 2000).

In this work an attempt has been made to estimate the sheet failure strength using a slightly modified equation (6.1) for DENT geometry. The fracture toughness estimated from cyclic technique was used as G_c and the width of the damage zone (w_d) was taken for the crack length correction. As facilities for measuring the damage zone were not available, it was assumed that it was equal to the length weighted fibre length, l as suggested by Kettunen and Niskanen (2000). The following simplified expression was used for the geometric factor, β for DENT geometrywithtotal crack length $2a$, width $2W$ (Yu and Karenlampi 1997);

$$\beta = \left[\frac{2W}{a}\left(\tan\frac{\pi a}{2W} + 0.1\sin\frac{\pi a}{W}\right)\right]^{1/2} \qquad \textbf{(6.2)}$$

Now a was replaced with $(a+w_d) = (a+l)$ to include a correction to the crack length. Then the apparent strength of a DENT specimen is then,

$$\sigma_{app} = \frac{\sqrt{w_c E}}{\beta\sqrt{2\pi(a+l)}} \qquad \textbf{(6.3)}$$

where w_c is the cyclic fracture toughness, expressed in J/m^2 and l is the length weighted fibre length. The elastic modulus, E, was estimated from a standard tensile test on un-notched specimen. The apparent strength of the sheets made from high coarseness radiata pine pulp refined for zero minutes to 75 minutes was estimated from this expression. The sample dimensions were kept constant and a ligament length of L =5.1 mm and a specimen width of $2W$ = 24 mm was used. The calculated value of β from equation 6.2 was 2.9 for the DENT geometry. Figure 6.10 shows the experimentally obtained failure stresses against the predicted apparent strengths for the different samples made from high coarseness radiata pine pulp. For each sample, the experimental failure stress was obtained from the maximum load divided by the sheet thickness and the width of the sample. Figure 6.10 shows that predicted values are well correlated with the experimental values. The average error in the predicted values was approximately 9%.

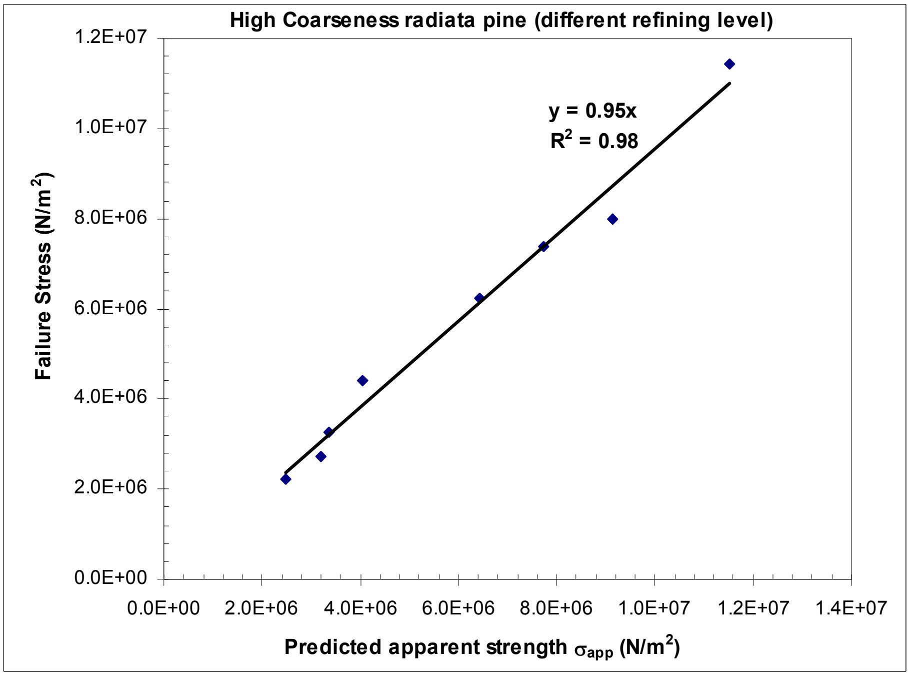
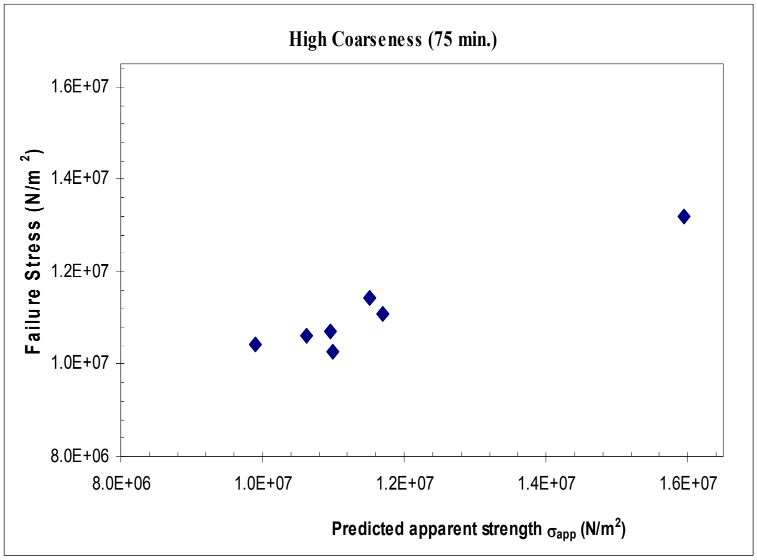
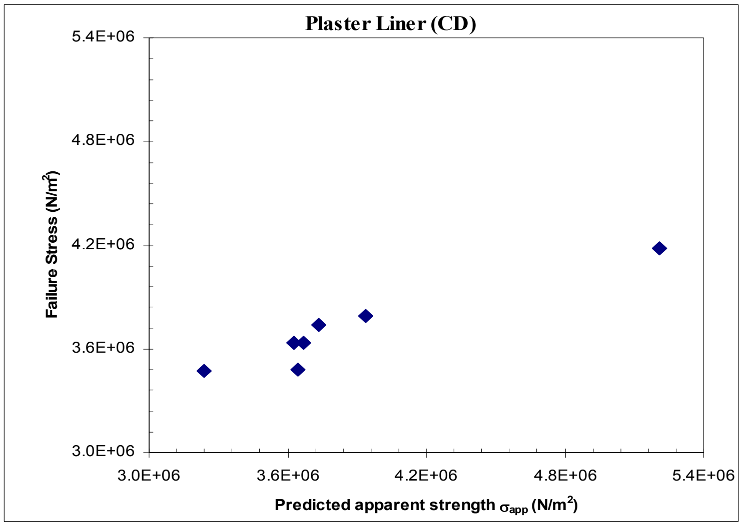


Figure 6.10. Failure stress against predicted apparent strength for the specimens made from high coarseness radiata pine refined between zero to 75 minutes

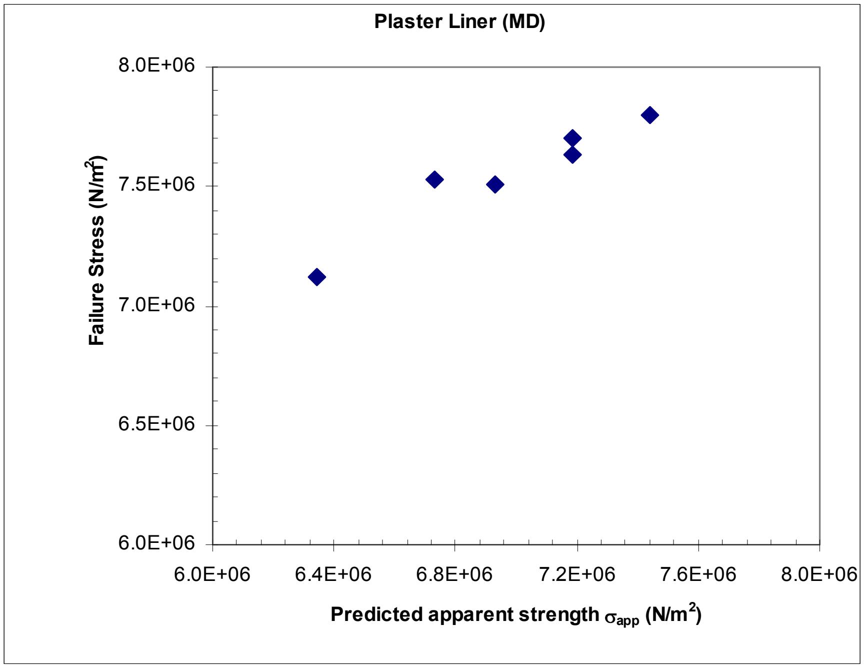
Further calculations were carried out to estimate the apparent failure strength on (1) high coarseness radiata pine specimens refined for 75 minutes, (2) commercial plaster liner boards in machine (MD) and (3) cross-machine directions (CD). This was conducted with the aim of testing the accuracy of the predictions when sample dimensions were varied. However the *a/W* ratio of these samples varied only slightly (*a/W*= 0.75 – 0.79) due to the reason that these dimensions were originally selected to satisfy the conditions in EWF technique. The length weighted fibre length for Plaster liner was assumed to be 1.8mm.



(a)



(b)



(c)

Figure 6.11. Failure stress against predicted apparent strength for (a) high coarseness radiata pine refined for 75 minutes (b) plaster linerboard tested in CD and (c) plaster liner in MD directions. Different data points in each plot represent the DENT specimen prepared at different dimensions.

Figure 6.11 shows the failure stress against predicted strength for samples made from (a) high coarseness radiata pine refined for 75 minutes (b) plaster liner board (CD) and (c) plaster liner board (MD). The predicted values were reasonably close to the experimentally obtained failure stress of these samples. The average error in the prediction of failure stress in samples made from high coarseness radiata pine refined for 75 minutes was about 11%. This error was higher for the largest ligament length, L=14.1mm, and this was observed for all 3 grades of samples. When L=14.1mm data point was removed the average error was reduced to 6% for the high coarseness sample. For plaster liner boards the average error was 11% for CD and 13% for MD. This error was reduced to 6% and 8% when L=14.1mm data was removed from the average error calculation for CD and MD respectively. The reason for higher error for the samples with L=14.1 mm was not clear. However, overall it can be concluded from these results that the substitution of fracture toughness values obtained from cyclic loading technique, in the expression shown in equation (6.3) estimates the sheet failure strength to a reasonable accuracy for these samples. Moreover, the accuracy of the measurements might have been improved if the damage width had been measured.

6.8 Conclusions

This chapter mainly presents a new cyclic method for the measurement of fracture toughness of paper. The fracture toughness measured using this new method has been compared with that measured from the Essential Work of Fracture (EWF) technique. It was found that the cyclic fracture toughness values were around 8% lower than those measured from EWF technique. For one commercial paper, tested in both MD and CD directions, and for one laboratory made sheet, the cyclic fracture toughness values were measured for ligament lengths from 3.3 to 14.1 mm and these values were compared with the EWF fracture measurements. The cyclic fracture toughness of the 2 toughest samples was generally independent of ligament length. However the weakest sample showed a small increasing trend in fracture toughness with ligament length.

The stability of the crack growth was also investigated by carrying out the cyclic fracture toughness measurements on 6 different gauge lengths ranging from 25 mm to 90mm and it was found that the effect of different test piece gauge lengths on fracture toughness measurements was insignificant. One of the assumptions from the literature is that the fracture must be stable in order to obtain accurate results. However, data presented in this chapter showed that the fracture can be partially unstable (brittle) without changing the measured fracture toughness and thus that the assumption from the literature is invalid.

The new cyclic technique has a number of advantages. This technique requires tests on test pieces of only one size and the total area required is only about 9 % of the sample area normally required for EWF measurements, thus significantly increasing the measurement speed. This new fracture toughness technique provides a significant improvement over the existing techniques. Table 6.1 shown below gives the estimated minimum time required for testing of FT of laboratory made paper using cyclic, EWF and STFI J-integral methods.

Table 6.1 The estimated minimum time for testing FT of laboratory made paper using Cyclic, EWF and STFI methods

	Sample cutting & preparation (minutes)	**Loading & Testing time per sample (minutes)**	**No of samples**	**Total time for loading and testing (minutes)**	**Other (minutes)**	**Total (minutes)**
Cyclic Method (using Instron)	3	1	12	12	-	15
EWF (using Instron)	60	1	60 (at least 5 different ligament lengths)	60	7 (plot graph & calculate FT)	127
STFI-J integral (using Instron)	3	0.5	30 (10x 15mm wide tensile and 20x50mm wide notched)	15	5 (to calculate maximum load & elongation)	23
STFI-J integral (L&W tester)	3		Same as with Instron	4		7

This chapter also gives the details of a study that was conducted to predict paper failure strength using sheet fracture toughness. The results revealed that the substitution of fracture toughness estimated from cyclic method into a relation derived from linear elastic fracture mechanics predicts the sheet failure strength at a reasonable accuracy. This accuracy might be improved if the damage width of the sheet fracture had been measured.