5 Comparison of fracture toughness of paper with tensile properties

5.1 Introduction

The importance of in-plane fracture toughness (FT) measurement of paper in terms of web runnability and quality control is well understood. However there are number of drawbacks in the use of currently available FT measurement techniques for routine measurements. The EWF method requires that a large number of samples of differing sizes be measured, consuming a significant amount of time to determine a single FT value. And also there are instances where the assumptions underlying the EWF technique are not always met (see Chapter 2 for more details). The use of multiple specimen J-integral technique creates number of problems in terms of convenience and reliability (Uesaka 1983b; Seth, Robertson et al. 1993). The rapidity and the ease of measurement has been subsequently considerably improved by the development of the J-integral tester (Welmar, Fellers et al, 1997) manufactured by L&W (Lorentzen & Wettre, 2004). However, the use of maximum load as the critical load to estimate the fracture toughness is not always correct (Tanaka, Otsuka *et al.* 1997).

The other main mechanical properties that are generally used in the paper industry as quality control (QC) measurements are the (in-plane) tensile properties, compression tests, tear and bending stiffness. The techniques for the measurement of the tensile properties of paper [tensile strength, elastic modulus and Tensile Energy Absorption (TEA)] are well established and straightforward. The basic similarity with both FT and tensile measurements is that samples in both tests are loaded in-plane to failure. If an accurate correlation between FT and any single or combined tensile properties could be established, then this could be used to predict the in-plane FT, and hence could ease some of the obstacles to the use of FT as a QC measurement that are currently being faced.

A previous correlation study has shown that FT strongly depends on tensile properties (Seth 1996). In particular, a strong relationship was found between FT and a combination

of tensile strength and extensibility of sheets made from a softwood pulp. In another study, conducted to find a correlation between web runnability and tensile properties in pressrooms a good relation was found between web break frequency and a combination of tensile properties (Uesaka, Ferahi et al. 2001).

The work reported in chapter is an effort to find a correlation between FT and individual tensile parameters and combined parameters. Various commercial papers and laboratory made hand-sheets have been used in this investigation. Details of sample preparation and experimental methods are given in chapter 3. The essential Work of Fracture (EWF) technique was used to evaluate the FT of paper.

5.2 Materials

The following commercial papers were used for the EWF and Tensile tests: (a) "Reflex" copy paper (MD and CD) (b) Plaster linerboard (MD and CD) (c) Sack kraft (MD and CD). Laboratory made hand sheets made of the following pulps were also tested; (d) Bleached *pinus radiata* (New Zealand), Ultra- low coarseness, lightly beaten (e) Bleached *pinus radiata* (New Zealand), medium coarseness, prepared at different beating and pressing levels. (f) Bleached *pinus radiata* (New Zealand) high coarseness, prepared at different beating levels. Further details of these pulps have been given in (Wahjudi, Duffy et al. 1998) and in Chapter 3.

Three levels of beating were used to refine the high coarseness *pinus radiata* pulp. The times allocated for the beating levels were 15, 30 and 75 minutes, to produce pulps labeled light, medium and heavily beaten, respectively. The Canadian Standard Freeness (CSF) of this pulp before refining was about 755 ml (Canadian Standard Freeness index). The freeness at the 15, 30 75 minutes beating levels were 725, 705 and 575 CSF, respectively.

Unbeaten, medium (30 minutes) and heavily beaten (60 minutes) medium coarseness *pinus radiata* pulps were also used for tests. The initial CSF of the pulp (before beating) was about 718 ml. After 30 minutes beating the CSF was reduced to 630 ml. The CSF of heavily beaten pulp was 460 ml. The handsheets were pressed at either low (2 bar) or high (6 bar) pressing pressure.

The initial CSF of the ultra-low coarseness pulp was 669 ml. This pulp was refined for 15 minutes in the Valley beater reducing the CSF to 631 ml.

All the samples were conditioned in accordance with the ISO 187 standard and all the test pieces were prepared and tested in the same standard atmospheric conditions. The span length of the DENT specimens was 90 mm.

5.3 Results and Discussion

5.3.1 EWF Results

5.3.1.1 Commercial papers

(i) Reflex copy (MD and CD)

Figure 5.1 shows representative load-extension curves obtained for different ligament lengths of 80 g/m² Reflex copy paper tested in the Machine Direction (MD).



Figure 5.1. Load –Extension curves obtained from Reflex copy (MD) DENT samples at different ligament lengths

The curve with the largest area under load-elongation plot is for L=14.1 mm, while the smallest represents the L=3.3 mm ligament length. The apparent thickness and density of these samples were 102 µm and 784 kg/m³ respectively. At least 15 DENT samples were tested for each ligament length *L*. The average area under each load-elongation curve was taken for the final w_f calculation.

To determine the FT (w_e) of the copy paper in the MD direction, the y-axis intercept of the data in Figure 5.2a must be obtained through extrapolation. However, the last 3 data points in the Figure 5.2a with L > 11 mm do not follow the same linear relationship as for points with 6 < L < 11 mm. As discussed in Chapter 4, the reason for the above behaviour is that when L>11 mm the sample has not completely yielded before the crack begins to propagate (Tanaka, Otsuka et al. 1997), which violates one of the basic assumptions in the EWF technique. Hence DENT test pieces with L>10 mm are not suitable to use with MD copy paper samples when applying the EWF method. A similar behavior was observed for CD copy paper samples, where the data obtained for $L \ge 17$ mm did not follow the same linear relation that was followed by the samples in the range $0 \le L \le 17$ mm. The points obtained, for MD measurement, from sample with ligament lengths $L \le 6$ mm appeared to be experiencing complete brittle failure. However, when a brittle failure occurs, then an overestimation of total work of fracture could be expected as the stored elastic energy is enough or greater than the energy required to complete the fracture. Since the observed work was less than expected, any influence from the brittle failure cannot be justified. A domination of plane-strain condition over plane stress on decreasing ligament could be another possible factor to give lower work at smaller ligament lengths. However this could be possible at a situation where the ligament length L < 3t (Levita 1996; Mouzakis, Karger-Kocsis et al. 2000), which was not the case for these samples. Therefore the reasons for low values of total work of fracture given at low ligament lengths were not clear. Complete brittle failure is defined in this study as the situation where the stored elastic energy is enough to completely fracture the sample once the crack has begun to propagate.

Another important argument that can be brought at this point on the existence of the second linear regression for L>10 mm is the way that plastic work could scale with the ligament length *L*. One of the pre-requisites for EWF technique is that the work in the

outer plastic zone should scale with the square of the ligament length ($W_p \alpha L^2$). When the shape of the deformation zone deviates from a circular/oval shape the condition W_p αL^2 is not necessarily satisfied. The presence of a second linear regression region with higher y-intercept (higher than the y-intercept of the first regression region) suggests that the plastic work in the second region (for $L \ge 10$ mm) may be scaling with both L and L^2 giving a relationship of the form $W_p = \beta_1 L t w_p + \beta_2 L^2 t w_p$. Thus an EWF plot of data in the second regression region will overestimate w_e by $\beta_l w_p$, while still maintaining linearity. The possibility of scaling W_p with L can be further justified by the fact that if sample has not fully yielded, before fracture occurs, then the plastic work may occur as the crack passes through the ligament. Since this plastic work is driven by the passage of the crack it will inherently tend to scale with L.

Figure 5.2b shows the w_f against *L* data fitted to a linear relationship for tests conducted on copy paper in the MD and CD direction. The data obtained for *L* less than 6 mm and greater than 11 mm for testing in the MD direction and $L \ge 17$ mm for the CD direction were omitted from the fitting.





The fracture toughness values determined from the y-intercepts in Figure 5.2b are 11.1 ± 0.4 J.m/kg in the MD direction and 8.2 ± 0.4 J.m/kg in the CD direction. The larger slope ($\beta w_p = 0.7$) for CD compared to that for MD ($\beta w_p = 0.3$) reflects the greater extensibility in CD direction.



Figure 5.2 (b). w_f against *L* for "Reflex" copy paper. The two straight lines are linear fits to individual MD and CD data sets.

(ii) Plaster liner board (MD and CD)



Figure 5.3. The w_f against L data sets and the respective linear fits to plaster liner for MD and CD tests.

Figure 5.3 shows the w_f against *L* data obtained for 190 g/m² plaster linerboard tested in MD and CD directions. It can be seen that, in contrast to the copy paper, both data sets are well fitted by a single straight line for all ligament lengths, indicating that the deformation fields. The estimated FT for plaster liner is 15.4 ± 0.3 J.m/kg for the MD direction and 8.4 ± 0.4 J.m/kg for the CD direction.

(iii) Sack kraft (MD and CD)

Figure 5.4 shows the w_f against *L* data for sack kraft tested in MD and CD directions. The estimated FT for the MD and CD directions are 21.6±0.6 J.m/kg and 18.5±0.8 J.m/kg, respectively.



Figure 5.4. w_f against *L* data for sack kraft along MD and CD. • - Sack kraft (MD) • - Sack kraft (CD)

5.3.1.2 Laboratory made sheets

(1) High Coarseness pinus radiata – Lightly, Medium and Heavily beaten

Figure 5.5 shows the plots of w_f against *L* for lightly, medium and heavily beaten high coarseness radiata pine. All these samples were only lightly wet pressed during sample preparation. A marked increase in FT with beating is clearly visible in the results and the

values for lightly, medium and heavily beaten samples are $w_e = 13.4$, 28.6 and 34.8 J.m/kg, respectively. The slope of the graphs (βw_p), which indicates the degree of plastic deformation (or extensibility) of each type of samples, has increased with increased beating level. The values of βw_p were 0.4, 0.6 and 0.8 J/kg respectively. In all three occasions the data points were well fitted to a straight line and didn't show any sign of deviation from linearity, indicating that the shape of the deformation field has remained constant.



Figure 5.5. w_f against *L* for high coarseness *pinus radiata* samples at Light, Medium and Heavy beating levels

(ii) Medium coarseness pinus radiata – Unbeaten

Figure 5.6 shows the data obtained for unbeaten *pinus radiata* for the two different pressing levels. The FT estimated for lightly (2 bar) pressed sample was only 6.6 ± 0.1 J.m/kg. The FT of highly pressed (6 bar) samples was slightly higher than that of lightly pressed sample and it was 7.3 ± 0.2 J.m/kg, an improvement of 11% over the lightly pressed sample.



Figure 5.6. w_f against *L* data for unbeaten medium coarseness radiata pine wet pressed *at* low and high wet-pressure

It is generally understood that in the process of wet pressing fibres undergo shifting, bending and conformation to better consolidate the sheet, which improves mechanical properties. However, a small increase in FT in the unbeaten medium coarseness radiata pine sample indicates that the level of pressing is insufficient to greatly improve mechanical properties. This is also consistent with the work of Page (1985), who found that wet pressing has much less influence than refining on the mechanical properties of paper made from previously dried pulp.



(iii) Medium coarseness pinus radiata - Medium beaten

Figure 5.7. w_f against *L* data for lightly and heavily pressed medium coarseness, medium beaten *pinus radiata*

Figure 5.7 shows that the FT of the medium beaten *pinus radiata*, which was 28.9 J.m/kg, was independent of the level of pressing used.

(iv) Medium coarseness pinus radiata - Heavily beaten

Figure 5.8 shows the w_f against *L* plots for the heavily beaten medium *pinus radiata*. The FT obtained for high and low wet pressing were very similar with 26.6 J.m/kg for low pressed samples and 26.9 J.m/kg for the high pressed samples.



Figure 5.8. w_f against *L* data for lightly and heavily wet pressed, heavily beaten *pinus* radiata. The straight lines are linear fits for the respective data sets

The difference in w_e of these two measurements is within the fitting uncertainties. The FT of the heavily beaten samples is ~8% less than for the medium beaten samples. The reduction in FT is likely due to a combination fibre shortening or fibre weakening due to excessive beating.



(v) Ultra-low coarseness *pinus radiata* – Lightly beaten

Figure 5.9. *w_f* vs *L* for lightly beaten, lightly pressed ultra-low coarseness *pinus radiata*

Figure 5.9 shows the plot of w_f against *L* for ultra-low coarseness, lightly beaten and lightly pressed *pinus radiata* samples. The high and medium coarseness pulps required at least 30 and 75 minutes beating time, respectively to produce a slope (βw_p) of about 0.8 J/kg, which the ultra-low coarseness pulp has reached after only 15 minutes of beating.

It is well understood that the action of beating chemical pulp like *pinus radiata* kraft pulp loosens the structure of the fibre wall and the surface resulting in internal and external fibrillation. This suggests that the thinner-walled ultra-low coarseness fibres (wall thickness 2.96 μ m) can be beaten more easily than the thicker-walled high coarseness fibres (wall thickness 3.57 μ m). More details on the fibre properties have been given in Chapter 3.

5.3.2 Tensile properties

Table 5.1 shows the tensile properties and fracture toughness values of all the test materials. For the commercial papers, the tensile index and elastic modulus in the MD direction were all at least 50% greater than in the CD direction.

| Sample | Comments | Tensile Index (N.m/kg) | Elastic Modulus (kN.m/kg) | TEA Index J/kg | Extension at maximum load (mm) | FT (Jm/kg) |
|----------------|----------|------------------------------|---------------------------------|----------------------|--------------------------------------|---------------|
| REFLEX | MD | 52.0±1.3 | 6.9±0.3 | 617±27 | 1.8±0.1 | 11.1 |
| СОРУ | CD | 34.4±1.3 | 4.4±0.2 | 1049±46 | 4.3±0.2 | 8.2 |
| PLASTER | MD | 58.1±1.1 | 6.2±0.1 | 802±44 | 2.0±0.1 | 15.4 |
| LINER | CD | 22.3±0.5 | 2.6±0.1 | 691±45 | 4.0±0.1 | 8.4 |
| SACK | MD | 63.9±3.7 | 7.0±0.3 | 793±65 | 1.8±0.1 | 21.6 |
| KRAFT | CD | 34.4±1.3 | 3.8±0.1 | 1004±139 | 3.7±0.5 | 18.5 |
| HC PINE LB | LP | 19.7±0.9 | 3.7±0.2 | 157±23 | 1.2±0.1 | 13.4 |
| HC PINE MB | LP | 46.0±2.0 | 6.0±0.2 | 785±96 | 2.4±0.2 | 28.6 |
| HC PINE HB | LP | 72.3±2.5 | 7.1±0.2 | 1883±104 3.8±0.1 | | 34.8 |
| MC PINE | LP | 14.9±0.5 | 2.9±0.1 | 85±4 | 0.9±0.1 | 6.6 |
| UB | HP | 19.0±0.3 | 3.4±0.1 | 143±9 | 1.2±0.1 | 7.3 |
| MC PINE | LP | 62.1±4.8 | 7.2±0.4 | 1480±29 | 3.3±0.5 | 28.9 |
| MB | HP | 64.6±4.9 | 6.7±0.3 | 1560±33 | 3.6±0.3 | 28.9 |
| MC PINE | LP | 69.1±2.6 | 6.8±0.3 | 1707±38 | 3.6±0.3 | 27.0 |
| HB | HP | 75.3±1.3 | 7.7±0.2 | 1726±32 | 3.3±0.2 | 26.6 |
| ULC PINE LB | LP | 44.3±1.3 | 5.4±0.2 | 984±42 | 3.1±0.2 | 19.2 |

Table 5.1 Summary of Tensile properties (L.Pres-Low pressing. H.Pres-High pressing

HC – High coarseness, MC – Medium coarseness, ULC – Ultra low coarseness
 HB – Heavily beaten, LB- Lightly beaten, UB Unbeaten
 LP- Lightly pressed, HP- Heavily pressed

The TEA index has shown some mixed results for commercial papers, as "Reflex" and sack kraft in the CD direction have shown larger TEA values than in the MD direction, in direct contrast to the FT results.

5.4 The correlation of FT and tensile parameters

FT was plotted against each tensile property to see the correlations. The data in each graph was fitted with a linear function forced through the origin.



Figure 5.10. Fracture toughness against TEA index. The relation showing y=0.0179x with $r^2=0.73$ is the linear regression fitted to all the data points. The other relation was obtained from fitting only data from the laboratory made paper samples.

The linear regression represented by y = 0.0179x ($R^2 = 0.73$) in Figure 5.10 gives the fitted relationship between FT and TEA index for the data points obtained for all 16 samples. When the linear fitting was carried out only considering the data points obtained from the laboratory made samples, the relationship obtained was y=0.018x with $R^2 = 0.83$. The significant improvement in R^2 when the linear regression was fitted with data points obtained from laboratory made samples indicates a better correlation of FT of laboratory made paper with its TEA. The poorer correlation observed, when data from all samples are tested, could be due to changes in the MD/CD ratio, which will affect the stress field and yielding behaviour around the crack tip.



Figure 5.11. FT against tensile index for machine made and laboratory made samples. The linear regression for all the samples is given by y = 0.4x with $R^2 = 0.70$. The linear correlation for only the laboratory made samples is given by y = 0.44x with $R^2=0.85$. The two circled points are from "Reflex" copy paper CD (left) and MD (right)

Figure 5.11 shows the correlation between FT and tensile index. The correlation between FT and tensile index was similar to that between FT and TEA. The R^2 was 0.70 for the fitting carried out for all 16 samples and when the machine made samples were omitted from the linear regression, the R^2 improved to 0.85. It was apparent that among the machine made samples the data points represent by "Reflex" copy paper are mainly responsible for weakening the correlation between FT and tensile index. If these samples were omitted, the R^2 improved to 0.76 and the average error was reduced to 18% from 27.7%.



Figure 5.12. FT against elastic modulus for all 16 samples measured. The linear regression for all the samples is given by y = 3.5x equation with $R^2 = 0.61$. The linear fitting for only the laboratory made samples is given by y = 3.99x with $R^2=0.83$.

Figure 5.12 gives the correlation between FT against elastic modulus. A similar trend to the previous correlations between laboratory made and machine made sheets was observed, where the correlation between FT and elastic modulus has improved once the machine made samples were omitted from the linear regression. The correlation obtained for all samples was FT = 3.5 (Elastic Modulus) with $R^2 = 0.61$ for all the samples while the correlation for the laboratory made samples alone was FT = 3.99 (Elastic modulus) with $R^2 = 0.83$.

The best correlation obtained so far with the FT versus tensile parameters was between FT and TEA index with $R^2=0.67$ for all 16 samples. When considering only the laboratory made papers, the best correlation was obtained from FT against tensile index with $R^2 = 0.85$. These results suggest that the machine made paper tested in here may have contained a wide range of fibres, originated from different wood species. The variation in MD/CD ratio, between machine made samples and in comparison to the handsheets, also could have affected the stress fields and yielding behaviour at the crack

tip. This means that it is likely that tensile parameters may not accurately predict the FT of samples in industrial environments where the sheets are made from fibres originated from different sources and where the MD/CD ratio will vary.

5.5 FT and combination of tensile parameters

Attempts were also made to find a correlation between fracture toughness and combinations of tensile parameters. Although paper is a non-linear, visco-elastic material, in this work an attempt was made to establish a correlation between the fracture energy w_e , and combinations of tensile parameters, chosen by assuming that the paper is a linear – elastic material. A relation between these parameters was obtained by estimating the work from an area under a linear stress-strain curve of an elastic material (Tanaka 2001). The approximate fracture energy of an elastic material under a stress-strain curve is $w_e = 1/2\sigma\varepsilon$, where σ and ε are stress and strain of the material at fracture respectively. Using Hook's Law, where the elastic modulus E is related to stress and strain by $E=\sigma/\varepsilon$, the relation $w_e = \frac{1}{2}\sigma^2/E$ can be obtained. At sample failure the critical stress is related to the tensile index of the material hence the stress was replaced by tensile index. When the FT estimated from the EWF method was plotted against (Tensile index)²/(Elastic modulus), it was found that a linear fit, with slope of 1, forced through the origin had an R² value of only 0.60. The correlation (R² = 0.63) was slightly improved when linear regression was fitted through laboratory made samples only.

Although it was expected that combined parameters might give better correlation than with any one single parameter, the observed correlation with these two combined parameters was not encouraging. However the relation was slightly modified by introducing *A*, *B* and *C* parameters to the above relation to obtain a relation $FT = A(Tensile Index)^{B}/(Elastic modulus)^{C}$. The "Solver" tool in Microsoft Excel was used to get the best *A*, *B*, and *C* parameters by minimizing the Sum Squares Error

 $[SSE = \sum_{i=1}^{n} (y_i - \hat{y}_i)^2]$ where the y_i 's are the actual values and the \hat{y}_i 's are the predicted

value from the regression] between the expected and estimated FT values for all 16 samples. Figure 5.13 shows the FT against $A(\text{Tensile Index})^B/(\text{Elastic modulus})^C$ for all 16 samples and for laboratory made samples alone. For all samples, the R² was improved to 0.67 and the *A*, *B* and *C* parameters obtained were 0.36, 1.25 and 0.50 respectively. When the data obtained from the laboratory made papers was fitted to a linear regression,

new *A*, *B* and *C* parameters were obtained. The new parameters were *A*=1.57, *B*=0.27, C= -0.91 and R² has improved to 0.88.



Figure 5.13. FT against A(Tensile Index)^B (Elastic modulus)^C. The correlation for all samples is FT= 0.36(Tensile Index)^{1.25}(Elastic modulus)^{-0.50} with R² = 0.67. The relation for laboratory made samples is FT = 1.57(Tensile Index)^{0.27}(Elastic modulus)^{-0.91}

One of the difficulties that arise in finding a correlation for FT with a combination of tensile index and elastic modulus is the strong intercorrelation between these two tensile parameters. Since these two parameters are not independent of each other, a significant difference in the obtained B and C numerical values can be seen when comparing with the fit to all samples and that of only with the laboratory made samples. This numerical variation is not intrinsic to the selected samples but is due to the inter-correlation in tensile parameters. Also the power coefficient of the elastic modulus (C) has been set by the program to a negative value to compensate for the contribution from tensile index, due to the high correlation between the two parameters. Figure 5.14 shows the plot of elastic modulus against 0.11(tensile index) for all the samples and this plot clearly shows the inter-correlation between elastic modulus and tensile index for all the samples.



Figure 5.14. Elastic modulus against 0.11 x (Tensile Index) for all the samples. A strong correlation can be seen between Elastic modulus and tensile index ($R^2 = 0.92$)

This means the use either tensile index or elastic modulus alone would be more appropriate in combination with other tensile parameters to estimate the FT.

Thus the next attempt at finding a multi-variable regression was to combine TEA with tensile index. The FT measured in the EWF technique is an energy. It estimates the work consumed per unit crack area in the FPZ. The tensile measurement TEA (Tensile Energy Absorption) is also a measure of energy that represents the total amount of mechanical work done on the entire sample up to failure. Although a better correlation between FT and TEA, compared to that with other tensile parameters, would be expected, the correlation shown in Figure 5.10 (FT versus TEA alone) does not give much support for this expectation. One reason could be due to a large part of the plastic work included in the TEA is irrelevant to the fracture. However, further attempts were made by combining tensile index and TEA and elastic modulus and TEA to see if any extra improvement in the correlation could be obtained.



Figure 5.15. FT versus $m(\text{Tensile Index})^n(\text{TEA})^o$



Figure 5.16. FT versus $p(TEA)^q$ (Elastic Modulus)^{*r*}

Figure 5.15 and 5.16 show the plots of FT versus combinations of tensile index-TEA and elastic modulus-TEA respectively. The *SSE* minimizing method was again used to find

the numerical variables that gave the best correlation between FT and the various tensile parameters.

The correlations obtained between FT against the TEA-tensile index and TEA-elastic modulus combinations for both the total data set and the laboratory made samples were close to each other in terms of *SSE* and R² values. The R² obtained for all the samples [FT versus $0.24(TI)^{0.55}(TEA)^{0.33}$ and FT versus $0.37(TEA)^{0.42}(E)^{0.63}$] was the same at 0.72. Here TI is the Tensile index and E is the Elastic modulus. The *SSE* values were 380 and 371 respectively. However R² values were improved to 0.87 and 0.88 when the machine made sample data points were omitted from the correlations. The correlations obtained then were FT = $1.16x(TI)^{0.25}(TEA)^{0.30}$ and FT = $1.47(TEA)^{0.21}(E)^{0.72}$] and the *SSE* were 111 and 102, respectively.

The possibility of combining all the tensile parameters to find a suitable multi-variable regression, that can further improve the correlation was also explored. However, the presence of strong inter–correlations in tensile parameters made the numerical parameters unstable and unreliable when predicting FT using such multi-variable correlations. As shown before in Figure 5.14 a strong correlation exists between Elastic modulus and tensile index. Not only that, but there are strong correlations between TEA and tensile index and elastic modulus as well.

Figures 5.17 and 5.18 show the relationships between Tensile index-TEA and Elastic modulus - TEA respectively. Tensile index has shown a fair correlation with TEA for all the samples, although the SSE was quite high. However, Tensile Index and TEA for the laboratory made samples were strongly correlated, with R^2 =0.98 and SSE=90. Elastic modulus has also shown a good correlation with TEA for the laboratory made samples with R^2 =0.96 and a very small SSE (SSE=1.23). Elimination of dependent parameters from the correlations will provide more stable numeric parameters in the multi-variable regressions. In that respect the combination of one tensile parameter, from the three interdependent parameters, and the other independent parameter, the extension at maximum load, will provide the best and most stable correlation.



Figure 5.17 Tensile index against $a(TEA)^b$ for all the samples ($R^2=0.73$) and only for laboratory made samples ($R^2=0.98$). A strong correlation can be seen for laboratory made samples.



Figure 5.18. Elastic modulus against $c(TEA)^d$ for all the samples (R²=0.53) and laboratory made samples (R²=0.96)

Hence it can be conclude that FT against *i*(tensile strength)^{*j*}(extension at max. load)^{*k*} should give the best correlation that can be obtained with independent tensile parameters. The use of this correlation is also supported by previous work in the literature (Seth,

1996) where this combination has shown an indirect relationship with EWF, TEA and burst strength under specified conditions.



Figure 5.19. FT against $i(TI)^{j}$ (extension at max. load)^k. The R² = 0.71 when the fitting was carried out on all the samples. The R² has improved to 0.87 when the machine made samples were omitted from the linear regression.

Figure 5.19 shows the FT versus $i(TI)^{j}$ (extension at max. load)^k plot for all the samples and only for laboratory made samples. There was no significant improvement in either correlation compared with previously obtained multi-variable numerical regressions. The *SSE* was 388 when the linear fitting was obtained for all the data points and R² was 0.71. The *i*, *j* and *k* parameters obtained in this fitting were 0.52, 0.85 and 0.32 respectively. Hence FT= $0.52(TI)^{0.85}$ (extension at max. load)^{0.32}. When the data from "Reflex" copy paper was omitted, the R² in the correlation improved to 0.81, the *SSE* fell to 207 and a new correlation of FT=1.08(TI)^{0.69}(extension at max. load)^{0.29} was obtained.

As seen in previous instances with other multi-variable numeric regressions, an improvement in R^2 was observed when only the laboratory made hand sheets were taken for the correlation (R^2 = 0.87, *SSE* = 115). The correlation obtained from this regression was FT= $1.72(TI)^{0.61}$ (extension at max. load)^{0.21}.

Thus although there has been a significant change in the numeric parameter *i* of the multi-variable regressions, the numeric power parameters associated for tensile index and extension (*j* and *k*) are reasonably stable in the three instances. This indicates that combination of tensile index and extension at maximum load has provided a more stable correlation than with the combination of independent and inter-dependent variables. Although the relationship between FT against $i(TI)^{j}(extension at max. load)^{k}$ is promising it appears that this relation works only well with selected grades and hence application of this correlation in the industrial environments may be unsuitable.

Table 5.2 summarises the R^2 , *SSE*, average and maximum errors obtained from different correlations. The errors were determined for each correlation by using the relevant fitted linear function to estimate the FT from the relevant tensile data. The FT estimated from the tensile property was then compared with the measured FT to determine the average and maximum errors for the data set. Among the individual tensile parameters, the correlation between FT-tensile index has shown an average error of 22% and a maximum error of 124%. It was apparent that the tensile values in the MD direction of "Reflex" copy paper and plaster liner were the least correlated with fracture toughness.

The relation $FT = i(TI)^{i}(Extension)^{k}$ provided the most stable correlation among all attempted correlations. The prediction of the FT using this correlation would produce an average 18 % error for the FT values of laboratory made samples with $R^2=0.87$. The maximum error in predicting FT that could occur from the use of this correlation is 40%. However, when the correlation was obtained for all 16 samples, the average error has increased to 28% and maximum error to 104%. The relationship FT= $i(TI)^{i}(Extension)^{k}$ is close to the relation obtained by Seth (1996) when he attempted to find a relationship between web break rate and in-plane tensile properties. The numerical parameters obtained for softwood pulps by Seth (1996) were i=1.08 j=0.63and k=0.52 compared to the numerical parameters i=1.72 j=0.61 and k=0.21 obtained for softwood kraft pulp in this work. Uesaka et al (2001) found a linear relationship, with a negative slope, between pressroom web break rate (%) and (MD tensile strength x extension^{1/2}) and this was approximately given by the equation, (Break rate %) = -0.65(MD Tensile strength x Extension $\frac{1}{2}$) + 2.0.

| | Samples | \mathbf{R}^2 | SSE | Average Error (%) | Maximum Error (%) |
|--|--------------------------------------|----------------|-----|----------------------|-------------------------|
| FT-TEA | All samples | 0.67 | 576 | 35 | 129 |
| | Laboratory made only | 0.83 | 385 | 31 | 78 |
| FT-Tensile Index (TI) | All samples | 0.66 | 458 | 22 | 124 |
| | Laboratory made only | 0.85 | 145 | 13 | 35 |
| FT-Elastic modulus (E) | All samples | 0.56 | 584 | 35 | 162 |
| | Laboratory made only | 0.83 | 150 | 24 | 86 |
| FT-A(TI) ^B | All sample | 0.65 | 454 | 28 | 126 |
| | Laboratory made only | 0.87 | 117 | 18 | 48 |
| $FT-m(TI)^n(TEA)^o$ | All samples | 0.71 | 380 | 21 | 103 |
| | Laboratory made only | 0.87 | 111 | 17 | 42 |
| $FT-p(TEA)^q(E)^r$ | All samples | 0.72 | 371 | 28 | 111 |
| | Laboratory made only | 0.88 | 102 | 15 | 41 |
| FT- <i>i</i> (TI) ^j (Extension) ^k | All samples | 0.70 | 388 | 28 | 104 |
| | All except "Reflex" copy paper | 0.81 | 205 | 18 | 64 |
| | Laboratory made only | 0.87 | 115 | 18 | 40 |

 Table 5.2 Summary of the correlation between fracture toughness and tensile properties

It might have been expected that of the individual tensile parameters, the best correlation would have been between TEA and FT, due to the fact that TEA is also a measure of energy. However in the CD and MD directions of "Reflex" copy paper and sack kraft the correlation with FT is extremely poor, as the TEA in the CD direction is higher than in the MD direction, but the FT of these samples behaves oppositely. As indicated earlier one reason for such behavior may be because of the use of a wide range of pulp furnishes, such as recycled paper, to make commercial sheets. Another reason is that the TEA includes contributions from the energy absorbed in fracture as well as plastic work in the rest of the sample. We cannot measure this plastic work directly, but it should be related to the slope, βw_p , determined from the EWF graph.

The values of βw_p for the data presented in this chapter have been plotted against the EWF FT in Figure 5.20. It can be seen that the correlation between plastic work (as estimated by βw_p) and FT is very poor even with laboratory made samples. This suggests that although FT and TEA both measure energy, the energy spent on the plastic work is not related to the FT and hence this plastic work, which is included in the TEA measurement, prevents a good correlation between FT and TEA.





As a final remark, these results indicate that the tensile properties of the samples tested here can be used to obtain a general idea about the FT of these materials. However the relationships between tensile properties and FT are not strong enough to predict the FT of the material. This highlights the requirement for a unique method, which can easily and rapidly evaluate the "intrinsic property" of fracture toughness.

5.6 CONCLUSIONS

Correlations between different individual tensile parameters and multi-variable tensile parameters were investigated to find the best correlation to predict FT. From the individual parameters FT-tensile index provided the best correlation with $R^2=0.85$ for the laboratory made samples. The average error in the use of tensile index to predict FT was

13% for laboratory made pinus radiata kraft pulp samples and 22% for all the tested papers in this study. The most stable correlation was obtained with the combination of tensile index and extension at maximum load where the use of relation FT= 1.72 $(TI)^{0.61}(Extension)^{0.21}$ predicted the FT values with an accuracy of 18% average error on laboratory made pinus radiata kraft pulp samples. However when this formula was applied to all the samples the predicted FT had a higher average error (28%). The discrepancy that was observed in the correlations of machine made and laboratory made samples used in this study could be mainly due to the variability in the MD/CD ratio of the machine made samples, where "Reflex" copy paper had the lowest value.

FT-TEA has shown a poorer correlation than expected probably due to the poor correlation between the plastic work, which is included in TEA, with FT. In general the correlations that were obtained between tensile parameters and FT were not strong enough to justify using measurements of tensile properties to predict the fracture toughness, especially in manufacturing or other converting environments. The much-needed technique for rapid evaluation of the FT of paper cannot be replaced by the use of tensile parameters. Therefore the requirement of a more reliable technique for rapid evaluation.