(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
19 February 2004 (19.02.2004)

PCT

(10) International Publication Number
WO 2004/015398 A1

(51) International Patent Classification[7]: G01N 3/34, 3/28, 19/08, 19/00, D21G 9/00

(21) International Application Number: PCT/AU2003/001000

(22) International Filing Date: 7 August 2003 (07.08.2003)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
2002950712 8 August 2002 (08.08.2002) AU

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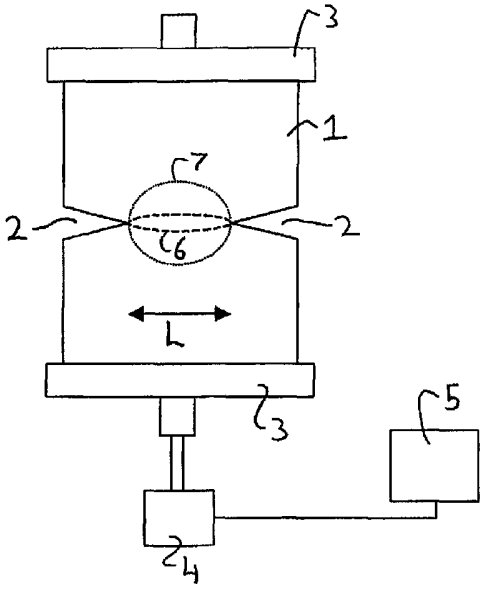
(81) Designated States *(national)*: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

**Published:**
— *with international search report*

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) **Title:** METHOD AND APPARATUS FOR DETERMINING FRACTURE TOUGHNESS




(57) **Abstract:** In order to determine fracture toughness of *e.g.* a sheet of paper (1), the sheet (1) is cut to provide a double-edge notched tension (DENT) geometry, with the notches (2) defining a ligament therebetween of length L. Load is applied to the sheet (1) by *e.g.* the movement apart of a pair of clamping elements (3) *e.g.* under the control of a motor (4) and controller (5), and extension versus load data is collected. The sheet (1) is cyclically loaded, *i.e.* loaded and unloaded a number of times, until it fractures, with the maximum applied load being increased on every cycle. The work done in the final cycle, in which failure occurs is calculated, and is used to provide the Essential Work of Fracture/Fracture Toughness for the sample (1).

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# Method and Apparatus for Determining Fracture Toughness

The present invention relates to a method and apparatus for determining fracture toughness. It relates particularly to the determination of fracture toughness of sheet material, and is especially useful in the determination of fracture toughness of paper stock and the like.

It is often important to be able to characterise the properties of a material, for example in order to determine the limitations of the material, how the material may be treated and/or used, the suitability of the material for a particular purpose and the like.

Various different parameters are used to provide a measure of these characteristics, and, for example, the tensile strength of a material may be found by loading a material until it breaks. This parameter may be useful when determining for example the suitability of a paper stock for use as a printing web in a printing process.

Another useful parameter is "fracture toughness". This is a measure of a material's resistance to crack propagation. It can be as important a parameter as e.g. tensile strength and the like, as materials are seldom perfect, and will often include defects (for example a paper web may include edge tears, pinholes, shives, end folds and the like). In a material lacking suitable fracture toughness, cracks may propagate from imperfections, and may cause failure long before the maximum stress suggested by the material's tensile strength is reached.

Thus, obtaining a reliable indicator of a material's fracture toughness can be very useful.

Two methods are currently employed in determining the fracture toughness of paper. These are the J-integral method, and the EWF (Essential Work of Fracture) method.

The J-integral method is based on non-linear fracture mechanics. In this method, tests are performed on an unnotched sample of paper in order to determine material behaviour. Thereafter, tests are performed on a notched sample, and fracture toughness is calculated by fitting data from the unnotched sample in order to obtain material parameters, and by finite element analysis techniques.

Various problems are associated with this method. For example, it requires testing of both notched and unnotched samples, there are difficulties in using it with tough paper, and some of the assumptions made by the technique are not always true. Also, in some cases, the estimated fracture toughness will depend on the sample or crack geometry, rather than being an intrinsic property of the material itself.

The EWF technique is an energy method that uses samples having a Double-Edge Notched Tension (DENT) geometry, i.e. a notch is made on both longitudinal side edges of a sample at the same point along the sample length. It assumes that the total energy to cause sample failure is consumed by both rupture at the crack and also by a plastic yielding zone around the crack, and that the first is proportional to ligament length (the length between the notches), whilst the second is proportional to the square of this length.

Accordingly, in the EWF method, a load-elongation curve is determined for a number of DENT notched samples of different ligament length, the total work done is estimated in each case from the area under the curve, and a plot is made of total work over ligament length versus ligament length. A straight line is then fitted to this data, and the fracture toughness is obtained from the y-axis intercept.

This method has some advantages over the J-integral method, and, for example, is independent of crack geometry. It does however have its own problems. These include the need to test a large number of samples of differing sizes. For example, a typical EWF determination may require the testing of 15 samples at each of 7 different ligament lengths. It can therefore be a time-consuming process, and is difficult to automate. Accordingly, it is difficult to apply this technique to quality control applications. It is also not ideal for research purposes, e.g. testing laboratory-made sheets, as the technique requires a large amount of sample material, which is time-consuming to produce.

The present invention aims to provide a new method and apparatus for determining a fracture toughness of a material, which looks to address the above problems.

Viewed from one aspect, the present invention provides a method of determining a fracture toughness for a material, including the steps of cyclically

loading a sample of the material, increasing the maximum load on the sample at each cycle until failure, and using work done in the cycle in which failure occurs to determine fracture toughness.

The present invention also provides an apparatus for determining a fracture toughness for a material, the apparatus including means for applying a load on a sample of the material in a cyclic manner, means for increasing the maximum load applied on each cycle, and means for recording load-extension data for at least the cycle in which the sample fails.

The present invention provides an elegant and simple method of determining the fracture toughness of a material, e.g. sheet material, such as paper or the like.

The method only requires the testing of a sample of a single size, and is quick and accurate and independent of sample geometry.

The method builds on the EWF method, and assumes that the work done in the final (failure) cycle may be equated with the Essential Work of Fracture, whilst the work done in the preceding cycles is not essential to the work of fracture (as there is no fracture propagation), and instead is the work done in the plastic yield zone. Experimental data obtained has indicated that this is indeed a valid premise, and further that the cycling of the material prior to failure does not introduce any significant complications caused by e.g. fatigue effects.

The invention is therefore based on the idea of loading a material up to a point below fracture, so as to exhaust the plastic deformation in a process zone around the crack tip, and to then reload the material to failure, the work done during the reloading being basically the essential work of fracture which can be used as a good parameter for fracture toughness.

For example, the essential work of fracture/fracture toughness may be determined to be the work done in the final cycle divided by the ligament length of the sample and the sample grammage.

The present invention provides a simple method of separating the Essential Work of Fracture (which is indicative of fracture toughness) from the work expended in the plastic region of the crack process zone, without the need to test a large number of samples, and without having to rely on the ligament length relationship assumptions of the standard EWF methodology.

As a sample need be measured only at one ligament length, the present invention can be conducted more quickly than the standard EWF method, which can be important for quality control applications. It also requires the use of less material, which can be important in research applications that use handmade paper or the like.

Further, as the fracture toughness can be determined independently at different ligament lengths, the independence of the result from sample geometry may be easily checked by comparing the results for different sample ligament lengths. This contrasts to the standard EWF method, in which it is only possible to check this independence by varying sample thickness. This feature also allows for example an average fracture toughness to be taken from measurements made at a range of ligament lengths.

Another advantage is that the technique is readily automated. For example, as a test may be carried out on a single sample size, an instrument can be purpose-designed to test samples of that one size. Also, the sample size may be the same as is used in tensile strength measurements, and so the same machine may be designed to carry out both tests.

The material under test may be of any suitable form, and preferably the invention is used in the testing of sheet material. The material itself may be of any suitable type, and the invention is particularly useful in the testing of paper products. The inventive technique may however also be used in the testing of other elastic-plastic or visco-elastic materials, and may be used in testing, e.g., polymers and/or metals. For example, it could be used to test polypropylene.

The number of cycles used in a test, and the amount of load increment between each cycle, may vary as required, and for example may depend on the material to be tested and the information already known about the material.

The test could begin with a relatively low load initial cycle, and increment in steps from there, or could begin with a relatively large load initial cycle and increment in steps from this higher value. The latter scenario would speed up the testing process, as less cycles would be needed, and could also reduce any possible fatigue effects and the like. The former scenario would however be useful for materials that might require a gradual increase in load in order to determine meaningful results. This would be true for example if the material under test had unknown stress-strain behaviour, as the maximum load would

then need to be incremented gradually. It would also be true if the material under test underwent large amounts of plastic deformation, in which case very small increments might be required so as to approach as close as possible to the point of maximum load before failure.

The load increment could be constant (e.g. a typical increment might be for example 1 N per cycle), or could vary between cycles. For example, the increment or increments could decrease in size, e.g. so that they would be larger in the first few cycles, and smaller in the final few cycles.

One object generally is to maximise the amount of plastic work that is carried out in the plastic yield zone, about the crack propagation zone, prior to the final failure cycle, so that the work done in the final cycle equates as closely as possible with the essential work of fracture. Thus, smaller load increments towards the end of the test can be advantageous, as they allow for a closer approach to the limit point where the plastic work ends and the essential work of fracture begins.

Preferably, the testing regime minimises the number of cycles used, so as to speed the testing process, and, for example, the test may include only two cycles, a first to perform work in the plastic deformation of the plastic yield process zone, and a second to perform the essential work of fracture. If the characteristics of the material under test are generally well-known, then this may be achieved by knowing, through experience, the maximum load that can be taken prior to fracture, and this amount of load may be immediately applied to the material in the first cycle. One further cycle based on a suitable load increment may then be all that is needed to cause failure, although two or more cycles may be preferred in order to approach the failure cycle more slowly and so remove as much plastic deformation work from the final cycle as possible.

In one preferred embodiment, in at least one of the non-failure cycles (which could be the first and only such cycle), the extension-load data is not merely monitored, but is also analysed in real-time, and the results are used to determine when to stop loading the material. For example, the maximum load in a cycle might be set by determining whether a crack has begun to propagate.

Thus, in one embodiment, loading is stopped when the material is detected to be undergoing a given plastic strain (plastic deformation or plastic flow). Thus, loading may be stopped when the material has undergone a plastic

strain of a set percentage, e.g. a 1% plastic strain. The actual percentage may vary depending on the type of material being tested, and may be set based on prior experience with the material. Other factors may also be taken into account, such as the rate of change of plastic strain and the like. These factors will indicate the extent to which the material is near to failure, and will allow the loading cycle to continue until just before failure.

This embodiment may facilitate the use of a two-cycle test, as it allows the first cycle to closely approach the fracture point of the material. Two or more cycles after the first cycle may also however be used if desired.

It should be noted that the plastic deformation sensed during the loading of the sample does not equate directly with the plastic deformation in the process zone around the crack, but rather is an average over the sample as a whole that can be used to infer when there has been complete or almost complete plastic deformation in the process zone of the crack.

Thus, the stress-strain curve of the material may be monitored during the first load cycle, and an increasing load may be continuously applied until the material reaches a non-elastic region of the curve.

Load may be applied to the material under any suitable control, and the control could for example be a load control and/or could be a displacement control.

In a displacement control, the extension of the sample is monitored, and loading is stopped when a set maximum extension is reached (which will correspond to some maximum load).

In a load control, the applied load itself is monitored, and loading is stopped when a predetermined maximum applied load is detected.

In one preferred embodiment, the test is applied under a mixture of displacement and load control, whereby the sample may be loaded at a fixed rate of displacement until the measured load reaches a set value, at which point the unloading process begins. This is preferable because it ensures that the sample is completely fractured in the final cycle. Thus, if the final cycle were controlled by a maximum displacement, and this were reached part way through fracture, then the testing instrument would stop applying load, and the fracture process would stop before it had ended. However, in load control, the final set maximum load will not be reached when failure occurs, and so the testing

instrument will continue to extend the sample looking for the maximum load, and the fracture process will continue to the end. The testing instrument stops loading once full fracture is achieved, e.g. by detecting when the measured load drops to zero.

Loading and unloading of the sample in each cycle may be achieved in any suitable manner at any suitable speed.

Preferably, in both load and displacement control, the sample is extended (to either the maximum set load or displacement) at a constant rate of displacement/extension (e.g. from about 2 to about 5 mm/minute). This rate of displacement may take any suitable value, and is preferably consistent between tests, as paper and other polymers are visco-elastic in character, and so the final fracture toughness result may depend on the loading rate. In one preferred embodiment, loading takes place at 2 mm/minute. It could also take place for example at the ISO specified standard for tensile testing, e.g. 5mm/minute.

Preferably, in the non-failure cycle or cycles, the material is also unloaded at a constant rate. This may be e.g. at the same rate as the loading rate, although, in some embodiments, the unloading rate may be higher than the loading rate, e.g. at 10 mm/minute. The higher rate has the advantage of increasing the overall speed of the test, and may be used e.g. in cases where data from the non-failure cycles is not used in the calculation of the Essential Work of Fracture, as will often be the case. In cases where non-failure data is used, e.g. as discussed below, then the unloading rate should be controlled with an eye to obtaining meaningful data for the unloading part of the cycle. Thus, the unloading rate may be the same as the loading rate and e.g. may be 2 mm/minute. Unloading will generally stop when the sample reaches zero load.

Where the material under test may be somewhat variable in character, e.g. paper or the like, the cyclic test may be performed on a number of samples, e.g. 10-15, and an average, such as the arithmetic mean, of the resulting EWF or fracture toughness values may be used to provide the final material fracture toughness.

The work done in the final cycle may be used itself as the Essential Work of Fracture, or may be modified, e.g. to compensate for remaining plastic work not removed in the pre-failure cycles. Remaining plastic work may for example become a significant factor in materials that exhibit a large amount of plastic

deformation. This is because it can be difficult in these cases to utilise small enough load increments to approach closely the point of failure of the material in the non-failure cycles.

In one preferred embodiment, the work done in the final (failure) cycle may be modified using the unloading part of the load-extension curve obtained from the second to last cycle, i.e. the final non-failure cycle.

Thus, the work done that is defined by the area under the final (failure) curve may be reduced by the work done in the area of overlap of the load-elongation curves of the final (failure) cycle and the preceding cycle. The area used to calculate the Essential Work of Fracture may thus be that bounded by the unloading portion of the final non-failure cycle load-elongation curve (or a straight line fit to this unloading portion) and the portion of the load-elongation curve of the failure cycle that extends onwards from the intersection with the final non-failure curve.

In a further possible modification to the work done in the final (failure) cycle, a slope may be fitted to either the loading portion of the final (failure) curve, or to the unloading portion of the second to last (final non-failure) curve, or to a combination of both, e.g. an average of the two slopes. A line with this slope may then be intersected with the final (failure) curve at the point of maximum attained load, and the portion of the final (failure) curve on the loading side of this line may be excluded from the final work done calculation.

In a still further possible modification, the unloading portion of the load-elongation curve of the second to last cycle is extrapolated to the maximum load obtained in the final cycle, and this portion (including the extrapolated part) is then displaced, so that the extrapolated maximum and the maximum of the final load curve are co-incident. The area bound by the displaced portion and the final load curve may then be used to determine the fracture toughness.

These embodiments again compensate for remaining plastic work, and can be especially useful in weak and high plastic deformation materials, where sufficiently small load increments between cycles may be difficult to achieve.

These modifications to the work done in the final cycle may be seen as concentrating on the work done in the unloading portion of the final curve where the material is fracturing.

The geometry of the sample may take any suitable form, and preferably, a double notch geometry is used (e.g. a DENT geometry). A DENT geometry is advantageous, as it confines the failure between the two notches. Other geometries are also possible however. For example the material may be notched only on one side, or, instead of notches, the material may be slit at one or more places across its width, e.g. the material may include a centre slit in the material perpendicular to the direction of loading.

In order to ensure proper working of the technique, the sample material preferably fulfils the conditions that: (i) the ligament is at least 5 times the thickness of the material; (ii) the ligament is equal to or less than a third of the width of the material; and (iii) the sample material will completely yield before crack propagation begins.

The present invention may be used in many different situations. Its simplicity renders it particularly suitable for research and quality control applications. It may for example be used in the quality control of a web, e.g. paper web, e.g. used in printing processes and the like, such as in producing paper rolls for newspaper printing. It may for example provide information as to the runnability of paper stock or paperboard or the like. It may be used in a paper mill, e.g. in an end-of-reel test. It may also be used in the quality control of packaging and of paper sacks and the like, e.g. paper sacks for storing cement, which need to cope with a high degree of rough handling e.g. during filling and transportation.

In order to ensure that the material under test is behaving as expected for valid results, the invention may allow for a check step. For example, when conducting a cyclic test, the work done during each cycle may be determined, and the total work done calculated. An identical sample may then be placed under increasing load until it fails without cycling, and the energy to cause failure may be compared with the total work done in the cyclic test. If the two values correspond, then the cyclic test may be considered applicable, as both values will be the plastic deformation energy together with the fracture energy.

A further check is to monitor the load versus extension gradient for each of the load cycles, and to compare them. If they are of similar values, then again this suggests that the test results will be valid.

Also, a check could be made by testing the material at a number of different sample ligament lengths, and checking that similar results are obtained.

Another check is that the sample does not undergo complete brittle failure. Complete brittle failure can be observed if, after fracture begins, the load-displacement curve displays an unstable, precipitous drop to zero load. Complete brittle failure will occur when the elastic energy, stored at fracture, is larger than the essential work of fracture. If complete brittle fracture occurs, then the work measured in the last cycle will be the elastic energy stored at fracture, and will be an overestimate for the Essential Work of Fracture. If complete brittle failure does occur, then the test may be repeated with a sample of shorter length (sample length being the length of the sample in the direction of load rather than the ligament length). A shorter sample length will reduce the overall amount of elastic energy stored in the sample. This will reduce the chances of the stored elastic energy exceeding the essential work of fracture, and so will reduce the likelihood of complete brittle fracture.

The apparatus for conducting the testing may take any suitable form, and may for example include a pair of clamping elements, e.g. line-type clamps, between which the sample may be mounted, the clamping elements being movable apart relative to one another, e.g. through movement of one or both of the clamping elements.

The clamping element or elements may be moved by e.g. an electric motor which may be controlled so that it extends the sample at a constant rate and/or so that the force changes at a constant rate. Other actuators, such as hydraulic actuators and the like could also be used.

Various tensile testing machines already exist which may be adapted to the present invention, with e.g. suitable programming of the clamp-controlling elements. For example, the method may be implemented using an Instron model 5566 Universal Testing Machine, and an add-on may be designed for this.

The apparatus may include cutting means for producing notches/slits in the material sample.

The same tensile testing machine may be used to conduct both a standard tensile test measurement and a fracture toughness measurement.

A testing process may take the form of firstly cutting the sample to provide e.g. the DENT geometry, and then loading the sample into a programmed testing instrument. The user may then select a fracture toughness test from the testing instruments' options, so that suitable software will be run. This software may then ask how many tests are to be performed and the type of sample or samples to be tested. After entering the information, the cyclic EWF test will be conducted on the first sample, and, once this is finished, the software will request that the next sample be loaded. After all of the samples have been loaded and tested, the software will display the final results in a suitable format.

Viewed from another aspect, the present invention may be seen as providing a method of determining a fracture toughness for a material, including subjecting the material to at least two loading cycles, a first loading cycle loading the material so as to produce plastic deformation in a process zone in the sample, and a final cycle loading the material to failure, wherein work done in the final cycle is determined in order to provide a fracture toughness.

The present invention may also be seen as providing a method of determining a fracture toughness for a material, including the steps of loading a cut sample of the material in a first stage until the material exhibits plastic strain, unloading the material, reloading the material in at least one further stage until the material fails, and determining the work done in the failure stage.

Viewed from a still further aspect, the present invention provides a method for determining a fracture toughness of sheet material, including the steps of cutting one or more notches or slits in the sheet material, subjecting the material to a load, releasing the load, subjecting the material to a higher load such that the material fails, and determining work done whilst the material was subject to the failure loading.

Viewed from another aspect, the present invention provides a method of determining a fracture toughness for sheet material, including the steps of providing a sample of the material with one or more defects, such as a notch or notches, therein, clamping the sample at two opposed ends, cyclically subjecting the sample to a load, the load of each cycle being larger than for the previous cycle, and determining work done during a final load cycle when the sample fails.

Viewed from a further aspect, the present invention provides a method of determining a fracture toughness for a material, including the steps of loading a sample of the material up to a point below fracture, so as to substantially exhaust plastic deformation in a process zone around a crack tip in the sample, and of reloading the material to failure, work done during the reloading being used to determine a fracture toughness.

It should be noted that any of the features of any of the above aspects of the present invention may be applied to any of the other aspects, and that the invention also extends to the determination of the Essential Work of Fracture of a material, without necessarily equating this with fracture toughness.

Embodiments of the present invention will now be described, by way of example only, with reference to the accompanying drawings. It is to be understood that the particularity of the drawings does not supersede the generality of the preceding description of the invention.

In the drawings:

Figure 1 is a schematic diagram of a material sample mounted in a fracture toughness testing machine;

Figure 2 is a graph of load versus extension for a cyclic loading test in accordance with one embodiment of the present invention;

Figure 3 is a graph of load versus extension for a cyclic loading test on samples of a material of different length, showing stable and brittle failure regimes;

Figure 4 is a graph of determined cyclic fracture toughness against sample length;

Figure 5 is a graph of load versus extension for cyclic loading tests on three samples of differing plastic characteristics, illustrating another embodiment of the present invention.

Figure 6 is a graph of work done versus ligament length for data obtained from a cyclic test in accordance with the present invention and from a standard EWF test;

Figure 7 is a graph of cyclic fracture toughness versus standard EWF fracture toughness as measured for a number of paper samples;

Figure 8 is a graph of fracture toughness versus ligament length for a plaster-liner board sample in a machine direction;

Figure 9 is a graph of fracture toughness versus ligament length for a plaster-liner board sample in a cross direction; and

Figure 10 is a graph of fracture toughness versus ligament length for a heavily refined, high coarseness radiata pine handsheet.

Referring to Fig. 1, in order to determine a fracture toughness for a sheet of material 1, e.g. paper, the sheet 1 is firstly cut so as to provide a double-edge notched tension (DENT) geometry, i.e. notches 2 are cut into the sheet 1 at opposite points along the length of the sheet edges so as to provide a ligament between the notches 2 of length L. It should be noted that the notches 2 are exaggerated in size for clarity.

The sheet 1 is clamped between a pair of clamping elements 3 that are mounted in a suitable testing machine (not shown). The testing machine may take any suitable form, and could, for example, include an Instron model 5566 Universal Testing Machine.

A load is then applied to the sheet 1, in an in-plane test, through movement of one or both of the clamping elements 3 such that they move away from one another. During this movement, load versus extension data is collected.

Movement of the clamping elements 3 may be controlled through activation of an electric motor 4, e.g. a stepper motor, under the instruction of a controller 5 of the testing machine.

For example, in one instrument, a lower clamp may be fixed in position through connection with a baseplate, whilst an upper clamp may be connected to a moving cross-head through a load cell. A stepper motor may then control the position of the cross-head, and by an accurate calibration between the number of steps of the motor and the cross-head movement, the testing instrument can accurately determine the position of the cross-head, whilst recording also the data from the load cell. The force required to displace the sample a given distance can then be calculated.

The control of the tensile load on the sheet 1 may be through a load control, in which the sample is loaded so as to achieve a target load, or may be through a displacement control, in which the sample is loaded so as to achieve a target extension.

In one method, the sample is loaded under displacement control, e.g. at a fixed rate of displacement, but the loading cycle is stopped when a given load is reached. This helps to ensure that the test is controlled to full fracture, and does not stop part way through a fracture.

In accordance with the present invention, the sheet 1 is cyclically loaded, i.e. loaded and unloaded a number of times, until it fractures. With each load cycle, the maximum applied load is increased.

The work done in the final cycle in which the sheet 1 fails is calculated, and is taken as the Essential Work of Fracture.

A normal unit for the Essential Work of Fracture is $J/m^2$, where $m^2$ represents a unit area of fracture surface. However, it can be difficult to determine exactly the thickness of paper samples, as their surfaces compress very easily. Accordingly, for paper, the sample thickness is generally replaced by grammage (mass per unit area – $kg/m^2$), so that the Essential Work of Fracture is given in units of Jm/kg.

Thus, in order to obtain the Essential Work of Fracture/Fracture Toughness, one will generally normalise the work done in the final (failure) cycle by dividing it by the sample grammage and by the ligament length.

The Essential Work of Fracture is the work done in propagating the fracture in a crack propagation zone 6 between the notches 2, and can be used as a measure of the fracture toughness of the sheet 1.

A typical graph resulting from the cyclic loading of a sheet 1 is shown in Fig. 2. It is for a sample of paper made from ultra low coarseness radiata pine (medium beaten – 2 bar press) having a DENT geometry with a ligament length of 5.1mm.

As can be seen, in each cycle the load is increased up to a set maximum and then decreased again to near zero. The loading and unloading is generally carried out at a constant rate. The unloading rate can be different from the loading rate, e.g. in cases where data obtained during unloading is not critical, unloading may be carried out at a higher rate than for loading so as to speed up the overall test procedure. The loading/unloading is generally controlled through displacement control, i.e. by monitoring the extension rate, irrespective of whether the target for the cycle is a set maximum load or displacement.

The loading and unloading rate may be for example 2mm/minute, although an unloading rate of e.g. 10mm/minute could also be used where unloading data is not critical. It would also be possible for example to use a loading rate at the ISO standard for tensile stress testing of paper, which is 5mm/minute. The minimum load reached in the unloading cycle is set to a small positive number, e.g. 1N, in order to avoid bending the sample

It is preferable that the loading (and preferably unloading) rate is consistent, as the results for the Essential Work of Fracture may otherwise vary due to the visco-elastic nature of paper and the like.

With each cycle, the maximum load is incremented by a set amount, e.g. 1 N, until the sheet 1 fractures (in this case at just under 18 N of force). In the final cycle, the specified load is never reached, as the sample fractures. The controlling software continues to increase the displacement at 2 mm/min until the sample has completely fractured, at which point the test is halted, e.g. manually or by the software noting a zero-load condition.

The work done in the final cycle is then calculated from the area A under the final curve FC, and is used as the fracture toughness.

A point to note is that, before fracture, the curve FC of the final cycle displays approximately the same elastic modulus E as the preceding cycles, which suggests that there are no significant fatigue effects at play. It should, however, also be noted that the sample has undergone significant plastic deformation during the preceding cycles, producing a plastic deformation at the start of the final cycle of about 0.2mm.

The plastic work done in all of the cycles prior to the final cycle FC is not work that is essential to the work of fracture (as fracture only occurs in the final cycle FC). Therefore, in the present technique, it is assumed that this work is the work done in an outer plastic zone 7 of the sample 1, and that only the work done in the final cycle FC includes the work essential for fracture.

Further, in the present invention, the work done in this final cycle may be equated with the Essential Work of Fracture itself, and so to a fracture toughness of the material 1, i.e. it is assumed that there is no significant plastic deformation work done in the final cycle.

Thus, through the cyclic loading, the present invention provides a simple and elegant method of separating the plastic deformation work done in the outer

plastic zone 7 from the Essential Work of Fracture, which is the work that causes the propagation of the fracture in the fracture zone 6.

It should be noted that if fracture were to commence before the final cycle, then this could provide incorrect results. This may however be detected by comparing the maximum load achieved in the final cycle with that of the preceding cycle. Thus, if premature fracturing occurs, then the maximum load achieved in the final cycle will be reduced compared to that in the preceding cycle. This arises from the reduction in the ligament length as the crack has begun to propagate. When this condition is determined to arise, the test results will be rejected.

Another point to note with respect to Fig. 2 is that the fracture in the final cycle is not completely stable, but displays partially brittle failure, that is the load-displacement curve drops precipitously and in an unstable manner after fracture commences, but the load on the sample does not fall to zero, and further energy must be applied to the sample to complete the fracture process. The unstable fracture process represents the consumption in the fracture zone of the elastic energy stored in the sample at fracture, whilst the tail thereafter arises from the fibres that make up the paper, as even after the crack has propagated through a section in the sample, unbroken fibres will remain bridging between the fracture faces. These fibres must then be pulled out against the bonds that hold them to complete the fracture process.

It has previously been thought that that Essential Work of Fracture techniques would only work with stable fracture processes, e.g. exhibiting a more gradual curve on fracture than the sharp drop of Fig. 2. However, the present invention has been found to work not only in fracture processes that are completely stable, but also in fracture processes that are partially brittle, as in the Fig. 2 example.

If the load were to fall in an unstable manner completely to zero and without the tail of Fig. 2 (complete brittle failure), i.e. the stored elastic energy was enough to fully fracture the material, then the energy measured in the final curve would be representative of the elastic energy stored at fracture, rather than the essential work of fracture, and the cyclic test would provide an overestimate for the fracture toughness. Thus, if this were determined to occur, the results would be rejected.

If complete brittle failure does occur, then a possible solution is to use a sample of smaller length (that is the sample length in the direction of the applied load, e.g. between a pair of tensioning clamps). A smaller sample length will reduce the amount of elastic energy stored at fracture, and so will reduce the likelihood of complete brittle failure.

Fig. 3 shows two representative cyclic load-elongation curves for sample lengths I and II of 90 mm and 25 mm respectively for samples made from high coarseness radiata pine refined for 75 minutes in a Valley beater. As can be seen, the 25 mm long sample shows stable fracture at all points, whilst the 90 mm sample shows a partial brittle failure.

Fig. 4 shows measured cyclic fracture energy as a function of sample length for samples of the same material as in Fig. 3, including samples at the 25 mm and 90 mm lengths, and confirms that the measured cyclic fracture toughness is independent of the sample length for this material, thereby showing that the present method of determining fracture toughness may be used either when stable or when partially brittle failures occur.

The present invention has a number of potential advantages over the standard EWF methodology. For example, the cyclic technique only requires the measurement of a sample at one sample size, and so can be faster. For the same reason, it requires the use of less sample material. This advantage can be magnified, as the results have been found to be generally independent of ligament length L, and so only a relatively small ligament length sample need be tested.

As all of the tests can be conducted at one ligament length, the cyclic method lends itself more readily to automation in both testing and cutting. Automation is also facilitated by the fact that the fracture toughness can be calculated directly from the area under the final loading curve FC. Further, the same machine may be used to conduct a tensile stress measurement as well as a fracture toughness measurement.

In order to ensure proper working of the technique, the sample material preferably fulfils the conditions that: (i) the ligament is at least 5 times the thickness of the material; (ii) the ligament is equal to or less than a third of the width of the material; and (iii) the sample material will completely yield before crack propagation begins. These are also standard EWF requirements.

Any number of loading cycles and sizes of load increments may be used in the testing, as is suitable for the sample under test, and a balance may be struck between a small load increment/large number of cycles regime, which may make the testing more accurate but longer, and a larger increment/less number of cycles regime, which may be quicker but less accurate. Also, the increments may be varied so that larger increments are used at the start of a test, whilst smaller increments are used when approaching failure.

In one preferred embodiment, the maximum loading in at least one of the non-failure cycles may be determined by analysing the load-extension data in real-time. Thus, the load-extension data may be monitored to determine when the sheet 1 has begun to exhibit a given plastic deformation, and the application of load in that cycle may then be halted.

This monitoring is able to load the material to close to where plastic deformation about the crack is exhausted, and where crack propagation occurs. It can therefore help to reduce the number of load cycles required to be performed (e.g. down to just a pre-failure cycle and a failure cycle), and can help to provide an accurate value for the measured Essential Work of Fracture, as it may reduce to a large degree any influences caused by remaining plastic deformation in the final failure cycle.

The application of load may for example be stopped when the material exhibits a certain percentage of plastic deformation or strain, e.g. a 1% deformation. For example, a reference slope E may be fitted to the load-displacement data corresponding to an elastic region of the material, and a local slope may then be compared to the reference slope as loading continues until the local slope varies by too great a degree from the reference slope. The rate of deformation or the like may also be used. In another method, the plastic strain may be estimated, and the loading may be stopped based on this value. For example, the reference slope, E, may be extrapolated from a measured (load,displacement) point to determine the extension at zero load, which is the plastic strain.

Although the full amount of work done in the final (failure) cycle may be used to provide the Essential Work of Fracture and so Fracture Toughness, this need not always be the case. For example, the amount of work done may be reduced, e.g. to take account of work done in the final cycle in respect of any

remaining ability of the sample to undergo plastic deformation in the plastic yield zone 7. Compensation for this extra plastic work done may be particularly useful when testing materials that exhibit high degrees of plastic deformation, where it may be difficult for the non-failure load cycles to approach the failure point closely, e.g. due to the small size of load increments that would be required between cycles.

Fig. 5 shows one method of taking this extra plastic work done into account. This figure shows the load-extension cycles for three samples, $S_1$, $S_2$, $S_3$, each being of the same well-beaten, medium coarseness material (radiata pine, 30 min. beat, 2 bar press), but each was stored and tested at a different relative humidity (10%, 50% and 90% RH), so as to provide samples of high, medium and low strength respectively.

As can be seen, the cycle curves for the weakest material $S_3$ are quite flat, and so the increments in load between cycles need to be low. It is therefore difficult to approach closely the failure point of the material without actually passing it.

Accordingly, the work done under the final curve FC will contain all of the Essential Work of Fracture, but may also include a significant amount of work done in plastic deformation of the plastic yield zone 7.

In one method of accounting for this, the unloading part U of the curve FC-1 of the second to last cycle, i.e. the last non-failure cycle, is extrapolated from the point of maximum load in that cycle so that the maximum load is extended to equal the maximum load attained in the final (fracture) cycle FC. A displacement offset D is then added to the unloading portion U so that the displaced unloading portion U' (including the extrapolated part) intersects with the final load cycle curve FC at the point M of maximum load attained in the final load cycle.

Rather than take the total work done under the curve FC as the Essential Work of Fracture, the Essential Work of Fracture is then taken as the work done in the area that is under the final curve FC and to the right of the displaced curve U'. The Essential Work of Fracture is thus decreased by the amount of work done under the final curve FC that is to the left of the displaced curve U', which may be taken as work done in plastic deformation.

This method of extrapolation effectively allows the Essential Work of Fracture to be measured as if the loading in the second to last cycle (i.e. the final non-failure cycle) had been stopped just before fracture occurred.

As shown in Fig. 6, the use of the curve U' to reduce the amount of work done is most significant in respect of the weakest sample $S_3$, and a similar line U" for the middle strength material $S_2$ reduces the final work done by a lesser amount. In respect of the strongest material, $S_1$, the method would have little effect at all.

As a variation on this method, instead of extrapolating the unloading portion U of the second to last cycle FC-1, a line could be fitted to the loading portion L of the final (failure) curve FC, e.g. in an elastic region of it. This line could then be displaced so as to intersect the final curve FC at the point M of maximum attained load, and the area under the final curve FC and to the right of the displaced line could be used to calculate fracture toughness.

As a further alternative, instead of displacing the curve U to U', the area of overlap of the final curve FC and the second to last curve FC-1 could be subtracted from the area under the curve FC. Thus, the Fracture Toughness is in this case is equated with the work done under the curve defined by the unloading portion U of the second to last cycle FC-1 (i.e. the last non-failure cycle) and the portion of the final curve FC that extends to the right (in the failure direction) from the intersection N between the two curves.

This compensation is based on the fact that the hysteresis between the loading and the unloading portions of a cycle's curve arises from the visco-elasticity of the paper. The area enclosed between the loading and the unloading curve portions can thus be thought of as representing the work done in visco-elastic dissipation and is not related to the fracture of the sample.

Experimental results for various commercially manufactured and laboratory-made papers are shown in Fig. 6-10. The handmade sheets were made from bleached and unbleached radiata pine pulp, unbleached eucalypt kraft pulp, unbleached eucalypt NSSC pulp (Neutral Sulphite Semi Chemical) and fibre blends prepared from different proportions of unbleached radiata pine/eucalypt kraft and NSSC/unbleached radiata pine. The bleached radiata pine pulps were the New Zealand pinus radiata market kraft and these were received as dried sheets in 3 different fibre coarsenesses (dry fibre mass per

unit fibre length). The unbleached pulps were never-dried pulps and were collected from an Australian pulp mill. The handsheets were made using a Moving Belt Former, which closely simulates the drainage of an industrial scale paper machine. Handsheets were made from unrefined pulps and pulps that had been refined in a Valley beater for up to 75 minutes. The wet sheets were pressed using a roll press at either 2 or 6 bar pressure. The effect of both refining and pressing is to improve the bonding between the fibres and thus to increase the fracture toughness.

The DENT samples were cut with a die designed to provide multiple ligament lengths from a 220x220 $mm^2$ area sheet. A hydraulic swing beam cutting press was used to stamp out uniform test pieces using this die. All of the samples were conditioned in accordance with ISO 187 standard at 23°C and 50% R.H., and then tested in the same conditions. Sample lengths were 90 mm.

Both the EWF and cyclic tests were carried out on DENT samples using a pair of clamps mounted on an Instron model 5566 Universal Testing Machine. The clamps were adapted from a clamp designed for EWF testing of paper samples, but with the linear guide rods removed so as to eliminate frictional effects on the cycling.

The sample widths were selected such that the ligament length was less than or equal to a third of the width of the sample. The cross-head speed of the Instron machine was set at 2mm/minute in loading and 10 mm/mm in unloading. The higher unloading speed decreased the time taken for each test.

Fig. 6 shows data obtained from both the standard EWF technique and the present cyclic testing technique, as practiced on one of the laboratory-made samples of ultra-low coarseness radiata pine, as used to provide the data of Fig. 2.

The straight line fit to the EWF data yields a y-axis intercept and so a fracture toughness of 19.2 ± 0.7 Jm/kg (N.B. $R^2$ in the graphs is an indication of the goodness of fit of the straight line to the data points).

In the cyclic test that was conducted at a ligament length of 5.1mm, the work done in the final (failure) cycle was calculated to be 18.2±0.9 Jm/kg, which is 1.0 Jm/kg less than the fracture toughness determined from the EWF test.

Significantly, they are equal between errors, which suggests that the present technique has merit.

An important point to note is that the total work done in the cyclic test (i.e. the sum of the work done in each of the load cycles including the final failure cycle) is equal within errors to the work done in the standard (non-cyclic) EWF test on the 5.1mm ligament sample. This gives a strong indication that the repeated load cycling of the present technique does not cause any significant fatigue damage, and that the results are not invalidated by changes in the sample caused by the cyclic loading.

Fig. 7 shows a comparison of standard EWF and cyclic fracture toughness data for tests on 8 commercially manufactured papers and 31 laboratory made handsheets. The fracture toughness determined by the standard EWF method ranged from 4 to 35 Jm/kg. The cyclic fracture toughness was around 8% lower that the standard EWF values. A straight line was fitted to the data set, and, after including estimated errors, the slope was determined to be $0.92 \pm 0.01$. To further investigate the reason why the cyclic fracture results were generally lower than the standard EWF results, the results from the two techniques were compared when practised over a number of ligament lengths on three further samples (plaster liner-board samples mounted in both the machine-direction and cross-direction, and a heavily refined, high coarseness, radiata pine handsheet). Ligament lengths in the range of 3.3 to 14.1mm were tested.

Fig. 8 shows the data for the MD direction plaster liner-board. The standard EWF technique gives a fracture toughness of 12.95 Jm/kg. The data for the standard EWF technique all lie close to the fitted line apart from the 14.1 mm sample. This is consistent with the 14.1 mm sample not displaying a type i plastic deformation field (approximately circular deformation fields formed before the sample fractures), which is necessary for the EWF technique to apply, and suggests that the deformation field in that case is of type ii or type iii (type ii having plastic zones that amalgamate to form a single deformation field after a point of maximum stress has been reached, and type iii being ones in which a plastic zone is never formed across the ligament and remains concentrated around the crack tips so that most of the work in the outer plastic zone occurs as the sample fractures).

The cyclic results are constant over the ligament lengths 4.0 to 10.8 mm, and give a fracture toughness of 12.4 Jm/kg for the three lowest of these results. The fracture toughness at the 12.1 and 14.1mm ligament lengths was 15.6 and 17.3 Jm/kg respectively. The latter is just under the total energy measured at the same ligament length for the standard EWF method (18.7 Jm/kg), and indicates the transition to a type iii deformation field. The anomalous value for the cyclic fracture toughness, at the lowest ligament length of 3.3 mm, is thought to arise from the intersection of damage zones around the crack tip. These damage zones arise from bond breakage and fibre fracture during the initial loading cycles.

Fig. 9 shows the data for the cross-direction plaster liner-board, and shows a gradually decreasing trend for the cyclic fracture toughness with decreasing ligament length for ligaments between 3.3 and 9.0mm. For the longer lengths, however, the measured results are approximately constant.

Fig. 10 shows the data for the high toughness handsheet. For this strong, tough material, the cyclic results are completely independent of length for the range tested. It is to be noted in this example that the average cyclic fracture toughness is 29.4 Jm/kg, whilst the standard EWF result is 34.6 Jm/kg.

This discrepancy cannot be because the final cycle includes a significant amount of work in the plastic zone 7, as this would produce a cyclic value that was higher than the standard value. This discrepancy is thought to be due to the work required to create damage zones around the crack tips, prior to fracture occurring. The work required to create these damage zones reduces the cyclic fracture toughness, compared to the EWF fracture toughness and is believed to be the reason why the cyclic fracture toughness is around 8% lower than the EWF fracture toughness.

The invention has applications in many areas, especially in the area of quality control, where it can be used to determine the fracture toughness of material used in printing webs or to make packaging or the like, e.g. paper sacks or the like. It would also be useful in research and the testing of handmade product.

The process may be readily automated. A testing process may take the form of firstly cutting the sample to provide e.g. the DENT geometry, and then loading the sample into a programmed testing instrument such as the Instron

machine. The user may then select a fracture toughness test from the testing options, so that suitable software will then run. This software may ask how many tests are to be performed and the type of sample or samples to be tested. After entering the information, the cyclic EWF test may be conducted on the first sample, and, once finished, the software may request that the next sample be loaded. After all of the samples have been loaded and tested, the software may display the final results in a suitable format.

It is to be understood that various alterations, additions and/or modifications may be made to the parts previously described without departing from the ambit of the present invention, and that, in the light of the teachings of the present invention, the invention may be implemented in software, firmware and/or hardware, e.g. for controlling a suitable testing machine, in a variety of manners as would be understood by the skilled man.

The present techniques are not limited to use with paper materials, but could also be used with other materials, e.g. elastic-plastic or visco-elastic materials. It may be applied for example to metals and/or polymers, and may be used with e.g. polypropylene sheets.

Further, the sheet material 1 may be cut in different geometries, e.g. using a single notch 2 or one or more slits within the body of the sheet 1, e.g. a central slit perpendicular to the direction of application of the tensile force.

Where a variable material, such as paper, is tested, a number of samples may be tested at the same and/or at different ligament lengths, and the results may be averaged or the like in order to provide the material fracture toughness.

**Claims**

1. A method of determining a fracture toughness for a material, including the steps of cyclically loading a sample of the material, increasing the maximum load on the sample at each cycle until failure, and using work done in the cycle in which failure occurs to determine fracture toughness.

2. The method of claim 1, wherein the maximum load is increased by a constant amount for each cycle.

3. The method of claim 1, wherein in at least one cycle load – extension data is analysed to determine when to stop loading in that cycle.

4. The method of claim 3, wherein the load-extension data is analysed so as to determine when the material exhibits plastic strain.

5. The method of claim 4, wherein loading is stopped when the plastic strain has reached a predetermined value.

6. The method of any preceding claim, wherein one or more of the cycles are controlled through a load-control.

7. The method of any preceding claim, wherein one or more of the cycles are controlled through a displacement control.

8. The method of any preceding claim, wherein the material is extended at a constant rate during loading.

9. The method of any preceding claim, wherein the material is extended at a constant rate during loading until a set load is reached.

10. The method of any preceding claim, wherein the sample is sheet material having a double-edge notched tension geometry.

11. The method of any preceding claim, wherein the material is a paper material.

12. The method of any preceding claim, wherein the work done in the cycle in which failure occurs is determined to be the fracture toughness.

13. The method of any of claims 1 to 11, wherein fracture toughness is determined by recording a load-extension curve for the cycle in which failure occurs, by constructing a straight line of set slope such that it intersects the load-extension curve in the region of maximum attained load, and by calculating the area under the curve bounded by the straight line and by the portion of the load-extension curve extending from the intersection of the straight line and curve.

14. The method of claim 13, wherein a straight line is fitted to a loading portion of the load-extension curve in the failure cycle, and the set slope is determined from the slope of the fitted line.

15. The method of any of claims 1 to 11, wherein a load-extension curve is recorded for the load cycles, and wherein the fracture toughness is determined by extrapolating the unloading portion of the load-extension curve of the cycle preceding the failure cycle to the maximum load attained in the failure cycle, by displacing the resultant curve such that the extrapolated maximum load is coincident with the maximum load attained in the failure cycle, and by calculating the area bounded by the displaced curve and the portion of the final load-extension curve that extends from the intersection of the displaced curve and the final curve.

16. The method of any of claims 1 to 11, wherein the fracture toughness is determined by calculating the area under a load-extension curve of the failure cycle minus the area of overlap of this curve with a load-extension curve of the preceding cycle.

17. Apparatus for determining a fracture toughness for a material, the apparatus including means for applying a load on a sample of the material in a cyclic manner, means for increasing the maximum load applied on each cycle, and means for recording load-extension data for at least the cycle in which the sample fails.

18. The apparatus of claim 17, including means for cutting the sample so as to have a double edge notched geometry.

19. A method of determining a fracture toughness for a material, including subjecting the material to at least two loading cycles, a first loading cycle loading the material so as to produce plastic deformation in a process zone in the sample, and a final cycle loading the material to failure, wherein work done in the final cycle is determined in order to provide a fracture toughness.

20. A method of determining a fracture toughness for a material, including the steps of loading a cut sample of the material in a first stage until the material exhibits plastic strain, unloading the material, reloading the material in at least one further stage until the material fails, and determining work done in the failure stage.

21. A method for determining a fracture toughness of sheet material, including the steps of cutting one or more notches or slits in the sheet material, subjecting the material to a load, releasing the load, subjecting the material to a higher load such that the material fails, and determining work done whilst the material was subject to the failure loading.

22. A method of determining a fracture toughness for sheet material, including the steps of providing a sample of the material with one or more defects, such as a notch or notches, therein, clamping the sample at two opposed ends, cyclically subjecting the sample to a load, the load of each cycle being larger than for the previous cycle, and determining work done during a final load cycle when the sample fails.

23. A method of determining a fracture toughness for a material, including the steps of loading a sample of the material up to a point below fracture, so as to substantially exhaust plastic deformation in a process zone around a crack tip in the sample, and of reloading the material to failure, work done during the reloading being used to determine a fracture toughness.

24. A method of testing a material, including the steps of cyclically loading a sample of the material, increasing the maximum load on the sample at each cycle until failure, and determining work done in the cycle in which failure occurs.

25. A method of determining the Essential Work of Fracture for a material, including the steps of cyclically loading a sample of the material, increasing the maximum load on the sample at each cycle until failure, and using work done in the cycle in which failure occurs to determine the Essential Work of Fracture.

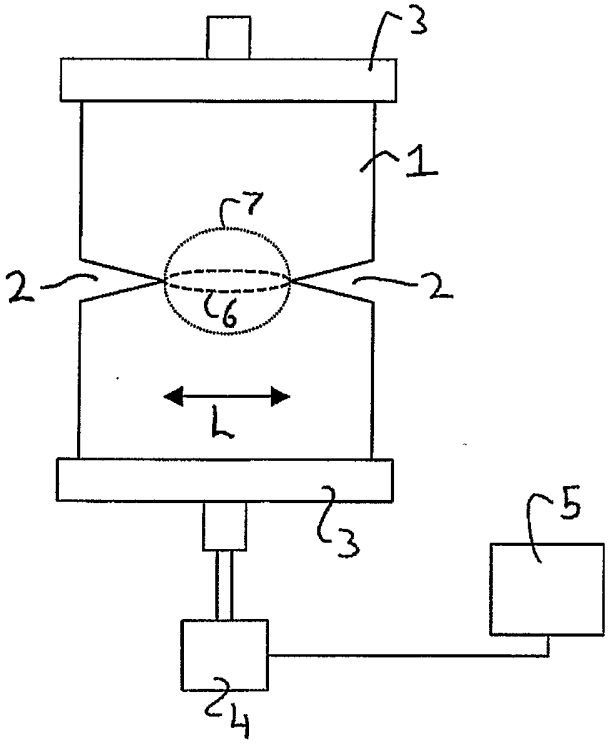



Fig. 1

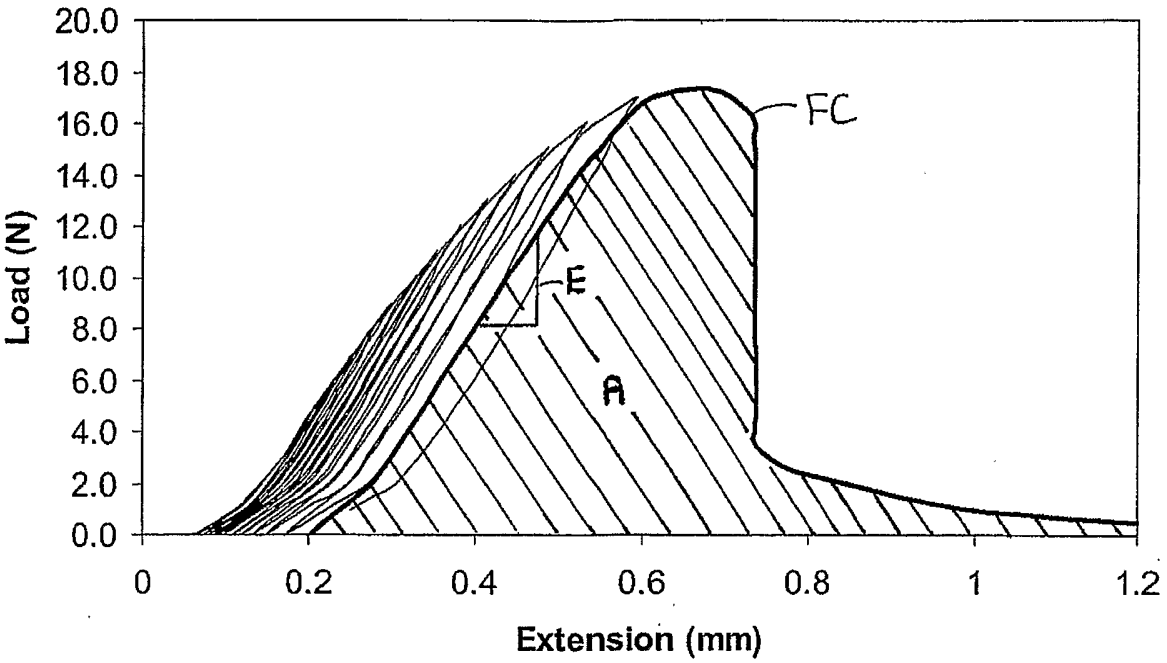

20.0
18.0
16.0
14.0
12.0
10.0
8.0
6.0
4.0
2.0
0.0
Load (N)
0
0.2
0.4
0.6
0.8
1
1.2
Extension (mm)
FC
E
A

Fig.2

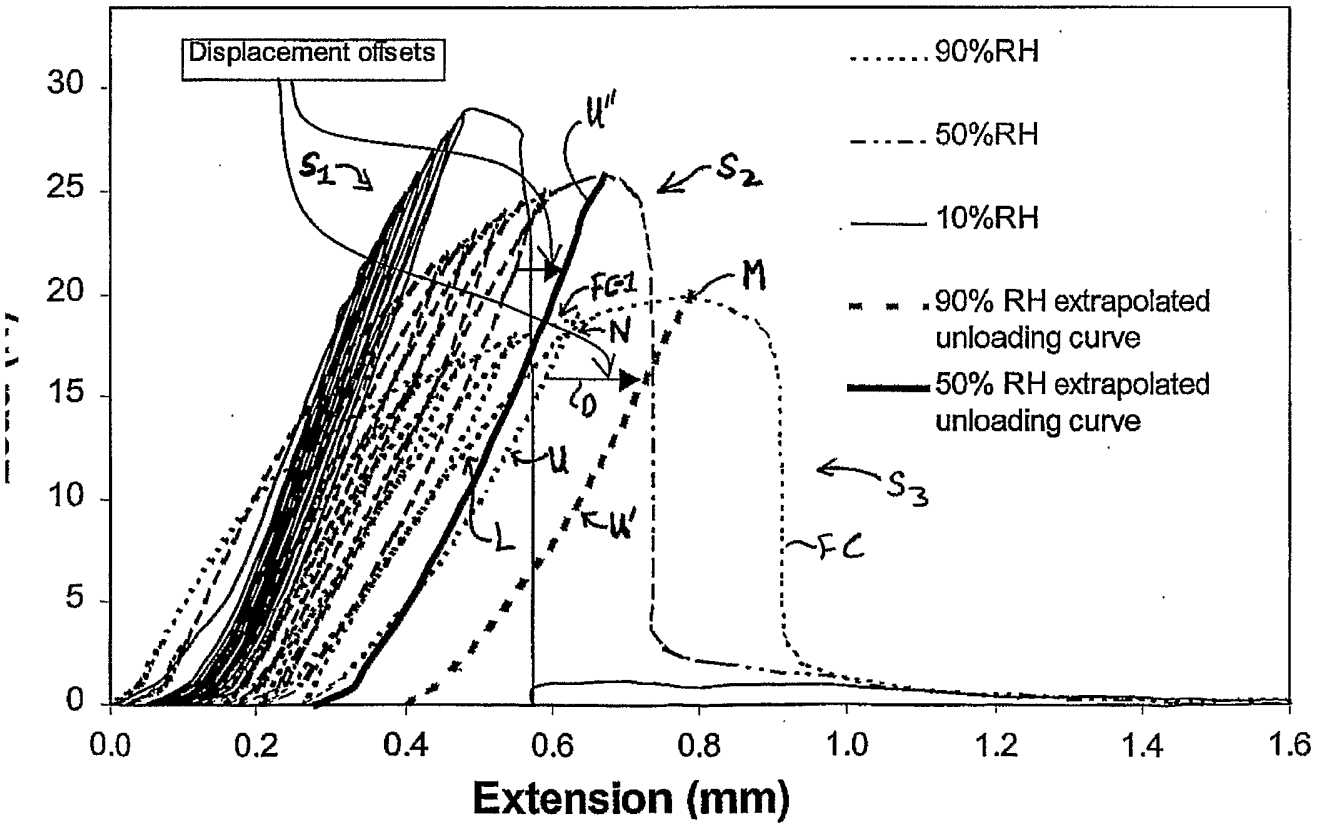

Displacement offsets
90%RH
50%RH
10%RH
90% RH extrapolated unloading curve
50% RH extrapolated unloading curve
30
25
20
15
10
5
0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
Extension (mm)
S1
S2
S3
U''
M
FC1
N
U
L
U'
FC

Fig.5

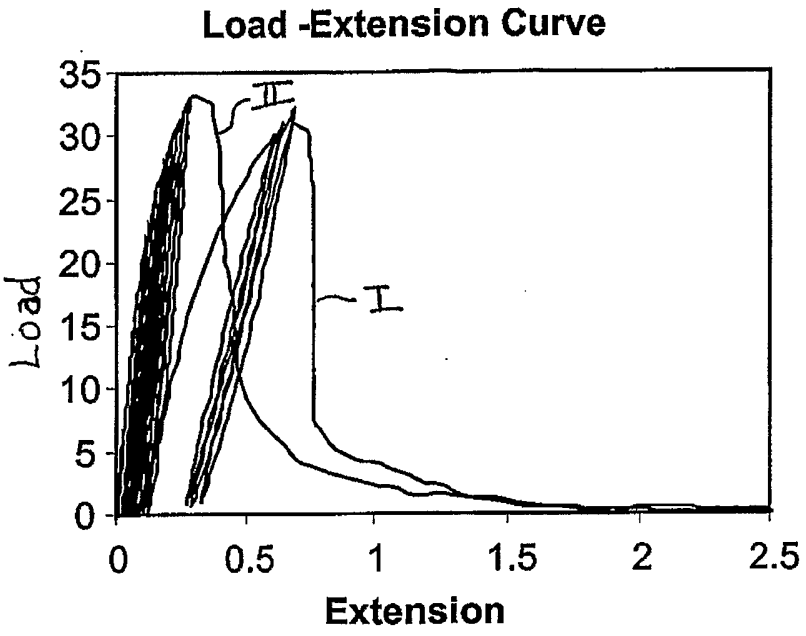

Load -Extension Curve
Load
Extension
II
I
0
5
10
15
20
25
30
35
0
0.5
1
1.5
2
2.5

Fig. 3

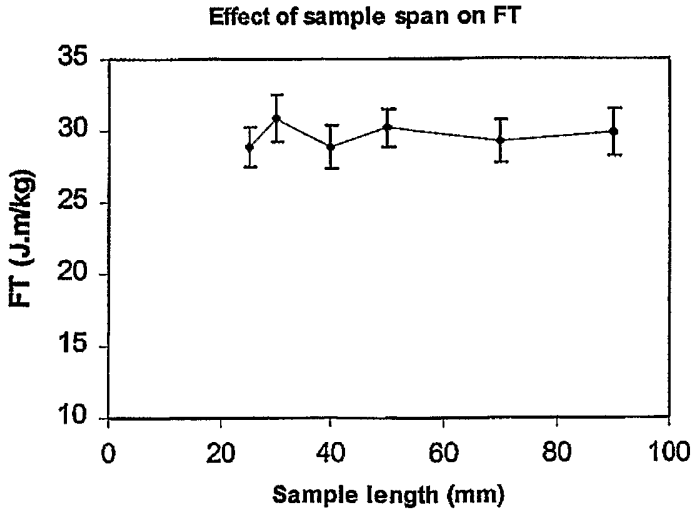

Effect of sample span on FT
FT (J.m/kg)
Sample length (mm)
10
15
20
25
30
35
0
20
40
60
80
100

Fig. 4

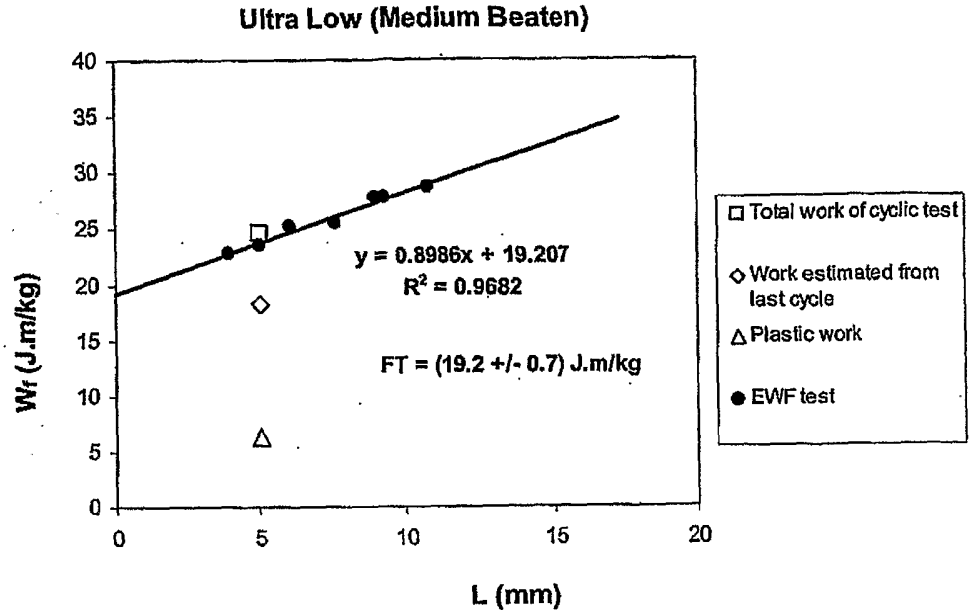

Ultra Low (Medium Beaten)
Wf (J.m/kg)
L (mm)
y = 0.8986x + 19.207
R² = 0.9682
FT = (19.2 +/- 0.7) J.m/kg
Total work of cyclic test
Work estimated from last cycle
Plastic work
EWF test

Fig. 6

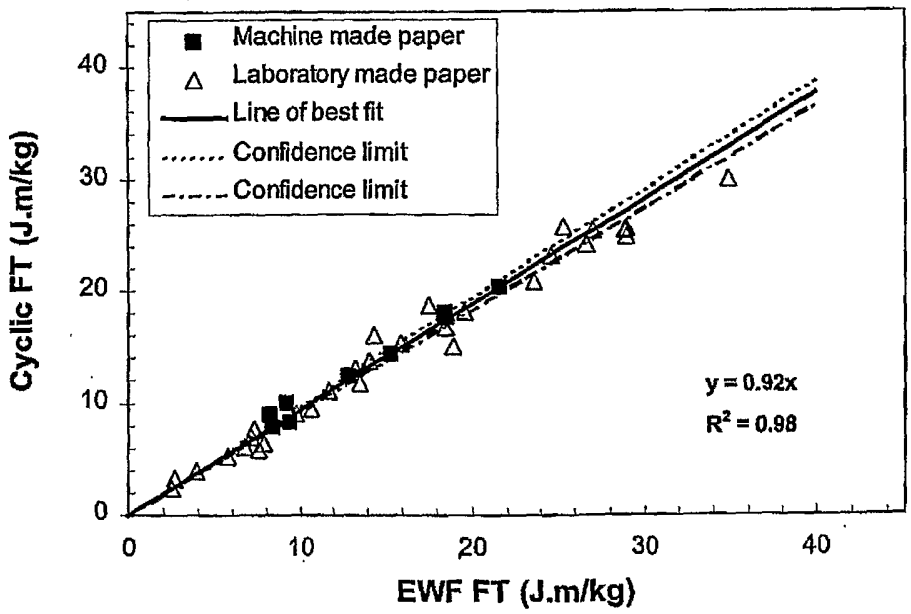

Machine made paper
Laboratory made paper
Line of best fit
Confidence limit
Confidence limit
Cyclic FT (J.m/kg)
EWF FT (J.m/kg)
y = 0.92x
R² = 0.98

Fig. 7

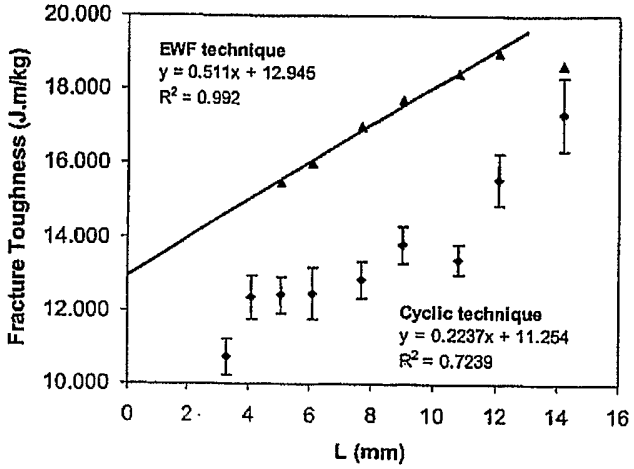



Fig. 8

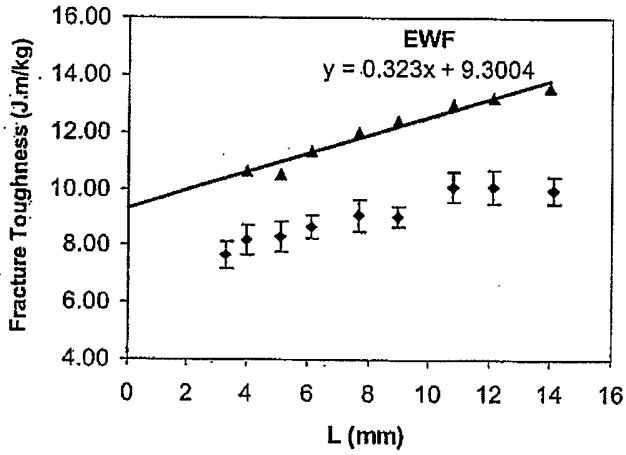



Fig. 9

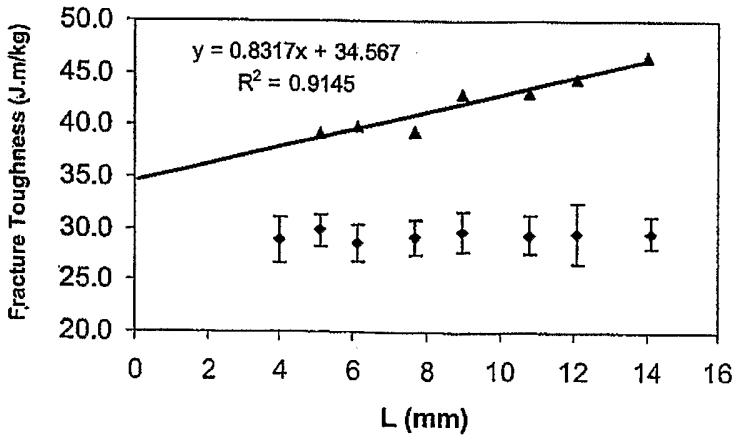



Fig. 10

# INTERNATIONAL SEARCH REPORT

International application No.
**PCT/AU03/01000**

**A. CLASSIFICATION OF SUBJECT MATTER**

Int. Cl. [7]: G01N 3/34, 3/28, 19/08, 19/00, D21G 9/00

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
DWPI: fracture (w) toughness, g01n/ic or d21g/ic, measur+ or test+ or determin+ or find+ or calculat+, method or process or procedure, cycl+ or again or repeat+ or reload+, fail+ or break+ or crack+, increas+ or bigger or higher or rais+ or larger, crack+ (2d) propagat+, plastic+ (2d) (strain or stress)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| P, A | US 2003/0041991 A1 (HAMAD et al.) 6 March 2003<br>Page 2, left column, lines 9-23; figure 2 | |
| P, A | US 2002/0174965 A1 (HAMAD et al.) 28 November 2002<br>Page 2, left column, lines 9-23; figure 2 | |
| P, A | WANIGARATNE et al., "Comparison of fracture toughness of paper with tensile properties", APPITA Journal, Vol 55, No. 5, September 2002, pages 369-374.<br>Whole document | |

[X] Further documents are listed in the continuation of Box C [X] See patent family annex

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 21 August 2003 | 29 AUG 2003 |
| Name and mailing address of the ISA/AU<br>AUSTRALIAN PATENT OFFICE<br>PO BOX 200, WODEN ACT 2606, AUSTRALIA<br>E-mail address: pct@ipaustralia.gov.au<br>Facsimile No. (02) 6285 3929 | Authorized officer<br>**RAJEEV DESHMUKH**<br>Telephone No : (02) 6283 2145 |

# INTERNATIONAL SEARCH REPORT

International application No.
**PCT/AU03/01000**

**C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| | US 4895027 A (MANAHAN, SR.) 23 January 1990 | |
| X | Figure 18; Description and claims | 1-25 |
| | US 4299120 (BARKER) 10 November 1981 | |
| X | Figures 4, 6, 7; Description and claims | 1-25 |
| | US 4116049 A (BARKER) 26 September 1978 | |
| A | Whole document | |
| | US 5602329 A (HAUBENSAK) 11 February 1997 | |
| A | Abstract | |
| | US 6053034 A (TSUI et al.) 25 April 2000 | |
| A | Abstract | |

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.
**PCT/AU03/01000**

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

| Patent Document Cited in Search Report | | Patent Family Member | | | | | |
|---|---|---|---|---|---|---|---|
| US | 2003/0041991 | NONE | | | | | |
| US | 2002/0174965 | NONE | | | | | |
| US | 4895027 | AU | 31980/89 | CA | 1325895 | WO | 8906790 |
| US | 4299120 | NONE | | | | | |
| US | 4116049 | NONE | | | | | |
| US | 5602329 | NONE | | | | | |
| US | 6053034 | NONE | | | | | |

END OF ANNEX