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**ÉTUDE DES PROPRIÉTÉS MÉCANIQUES DE LA LIAISON FIBRE-FIBRE
DANS LE PAPIER**

Abdellatif EL MAACHI

**DÉPARTEMENT DE GÉNIE PHYSIQUE
ÉCOLE POLYTECHNIQUE DE MONTRÉAL**

**THÈSE PRÉSENTÉE EN VUE DE L'OBTENTION DU GRADE DE
PHILOSOPHIAE DOCTOR (Ph. D.)**

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Cette thèse intitulée:

**ÉTUDE DES PROPRIÉTÉS MÉCANIQUES DE LA LIAISON FIBRE-FIBRE
DANS LE PAPIER**

présentée par: EL MAACHI Abdellatif

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a été dûment acceptée par le jury d'examen constitué de:

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À mes parents, à mes frères et sœurs

et à ma fille

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SOMMAIRE

Le pelage du papier a été utilisé afin de déterminer la ténacité de la liaison interfibre. Il a été supposé précédemment que toute l'énergie se dissipe dans la zone de détachement et est attribuée à l'énergie de détachement, et plus spécifiquement à la cassure des liaisons interfibre dans la zone de séparation. Cependant, dans ce travail, nous avons montré et quantifié les deux contributions énergétiques impliquées dans le pelage du papier, à savoir, l'énergie de détachement, W_a , et l'énergie de déformation, W_d . L'étude se compose de deux parties:

- (a) une étude expérimentale qui vise à déterminer l'énergie de pelage, W_p , et l'effet des paramètres influents, tel que l'angle la vitesse...
- (b) une étude théorique qui vise à décrire et modéliser le processus de pelage du papier.

L'étude expérimentale consiste à réaliser des échantillons de laboratoire laminés "laminated handsheet" à partir de la pâte kraft raffinée au degré désiré et dont l'épaisseur des deux couches de l'échantillon varie selon l'objectif de l'étude voulu. D'autres types d'échantillons (non laminés) ont été réalisés pour les tests de traction et compression dans le plan. Le pelage a été réalisé à des angles allant de 90° à 170° et à des vitesses allant de 0.5 à $25 \text{ cm}\cdot\text{min}^{-1}$. Les résultats de mesure ont montré que l'énergie de pelage, W_p , croît avec l'angle, la vitesse et l'épaisseur de la couche délaminiée (supérieur). Cependant, W_p est indépendant de l'épaisseur de la couche inférieure et décroît avec l'application d'un

ruban adhésif sur la couche supérieur. Ceci nous a conduit à suggérer l'implication de deux types d'énergies: L'énergie de détachement W_a , (dissipée dans la zone de détachement) et l'énergie de déformation, W_d (dissipée dans la propagation de la flexion).

L'étude théorique consiste à calculer W_d , en utilisant un modèle théorique basé sur les caractéristiques expérimentales contrainte-déformation en traction et en compression. Les résultats ont montré que W_d est pratiquement une fonction linéaire de la courbure de la flexion et que W_a est indépendante de la courbure (donc de l'angle). Nous avons montré de plus que, pour des échantillons laminés, W_a est indépendante de l'épaisseur, mais que W_a et la rigidité à la flexion affectent ρ_{\max} , et par la suite W_d . L'effet de W_a sur ρ_{\max} produit un phénomène d'amplification qui peut expliquer la sensibilité du test de pelage et les grandes fluctuations de la force de pelage observées expérimentalement.

L'effet du raffinage sur le travail de pelage et ses contributions (l'énergie de déformation et l'énergie de détachement) a été étudié. L'évolution de l'énergie de pelage et de l'énergie de déformation avec le raffinage présente un maximum, alors que l'énergie de détachement croît avec le raffinage. La variation de ce dernier a été attribuée à l'accroissement de la surface total de la liaison interfibre. Les résultats de l'énergie de déformation sont en bon accord avec les prédictions théoriques basées sur le modèle. Ces résultats nous ont permis d'attribuer la décroissance de W_p et W_d pour les hauts degrés de raffinage à l'endommagement prononcé des fibres et à la détérioration des propriétés mécaniques des fibres en traction et compression.

Abstract

Paper peeling or delamination is used to determine the toughness of fibre-fibre bonds. It has previously been assumed that all the work of peeling goes into the breaking of fibre-fibre bonds in the detachment zone between the fixed layer and the detached layer. In this work, we have demonstrated the contribution of the energy of deformation to the work of peeling. We present a method for quantifying both the energy of detachment and the energy of deformation involved in paper delamination. We have developed a theoretical model which describes the delamination process in paper. We have experimentally determined the effect of peeling parameters (angle and speed) and paper properties (thickness, beating level) on the work of peeling.

The peel test was performed on laminated handsheets at peel angles between 90° and 170° , and at rates between 0.5 and $25 \text{ cm}\cdot\text{min}^{-1}$. The results show that the work of peeling, W_p , increases with peel angle, speed, and the thickness of the top (detached) layer. However, W_p is independent of bottom layer thickness and decreases when adhesive tape is attached to the top layer. We conclude that paper undergoes inelastic peeling. This means that W_p is the sum of the energy spent on detachment in the detachment zone (work of detachment), W_a , and the energy spent for bending propagation (work of deformation), W_d .

In the theoretical model, we analyse the bending and unbending involved in flexion propagation during peeling, taking into account compressional and tensional strain-stress characteristics. The results of the calculation show that W_d is approximately a linear function of the curvature and that W_a is independent of angle.

A further analysis shows that W_a is independent of the basis weight (thickness) of laminated handsheet, but both W_a and the stiffness affect ρ_{\max} and, therefore, change W_d . The effect of W_a on ρ_{\max} is shown to produce an amplification phenomenon which can explain the high sensitivity of the peel test and the experimentally observed large fluctuation of peel force.

The effect of the beating of fibres upon the work of peeling of paper and its components (the work of detachment and the work of deformation) was investigated. The work of peeling and the work of deformation both show maxima as a function of the extent of beating. But the work of detachment increases with beating, which is due to the increase of the bonded area. The dependence of the work of deformation on beating agrees well with that predicted deformation work from the model based on in-plane tensile and compressive data. The decreases of both the work of deformation and the work of peeling at high beating level may be attributed to the extensive modification of fibres, which reduces their mechanical properties in tension and compression.

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CHAPITRE 1
INTRODUCTION GÉNÉRALE

1. INTRODUCTION et OBJECTIFS.

Le papier est un matériau hétérogène à tous les niveaux de sa structure. Il ne présente pas un continuum, comme c'est le cas pour les métaux et les polymères. Il en découle que les relations qui corrélient structure et comportement mécanique des métaux ou polymères synthétiques, ne sont pas directement applicables au papier. Cependant, dans plusieurs cas, le papier se comporte sous l'effet d'une déformation mécanique comme un corps continu.

Le papier est composé de fibres de cellulose (de diamètre moyen de $7\mu\text{m}$ et de longueur moyenne de 1 à 2 mm) disposées dans un réseau aléatoire à deux dimensions. A l'échelle moléculaire, la liaison fibre-fibre est basée sur les ponts d'hydrogène. La liaison fibre-fibre et les propriétés de la fibre sont des paramètres de première importance dans la fabrication du papier et l'utilisation pour laquelle il est conçu: impression, emballage, isolation diélectrique....

Les propriétés mécaniques de la liaison fibre-fibre et leurs effets sur celles du papier ont fait l'objet de plusieurs travaux expérimentaux et théoriques. Cependant, la dépendance des résultats sur des paramètres tels que la concentration de la contrainte et les différents traitements subis par les fibres, complique l'analyse des résultats et la détermination de la contribution des liaisons. En effet, il n'existe pas une technique

expérimentale capable de déterminer toutes les propriétés mécaniques de la liaison fibre-fibre. Plutôt, c'est un ensemble de tests qui est utilisé pour caractériser ces propriétés de façon approximative.

Les objectifs de cette thèse sont la description du pelage du papier et la caractérisation des différents processus et paramètres qui contrôlent la dissipation d'énergie.

1.2. STRUCTURE et PROPRIÉTÉS MÉCANIQUES des FIBRES et LIAISONS INTERFIBRES DANS LE PAPIER.

Le papier est un matériau très hétérogène en forme de feuille. Il est constitué de fibres de cellulose disposées dans un réseau plan. Son hétérogénéité s'étend de l'origine et la nature des fibres en passant par leurs dimensions et leur disposition dans la feuille (formation et distribution des parties liées et non liées des fibres) jusqu'aux contraintes figées dans la feuille ainsi formée. Cependant, dans plusieurs circonstances, le papier se comporte comme un corps continu.

L'utilisation du papier nécessite une amélioration constante de sa qualité et une adaptation à l'utilisation à laquelle il est destiné (impression, emballage...). Ceci nécessite la compréhension de son constituant élémentaire, à savoir la fibre, et ses interactions avec son environnement dans la feuille (liaison interfibre).

1.2.1. Composition et structure de la fibre et la liaison interfibre.

1.2.1.1. Structure de la fibre

La figure 1.1 présente une coupe transversale d'une cellule caractéristique des fibres. Chaque cellule est composée d'une paroi primaire (paroi externe, désignée par la

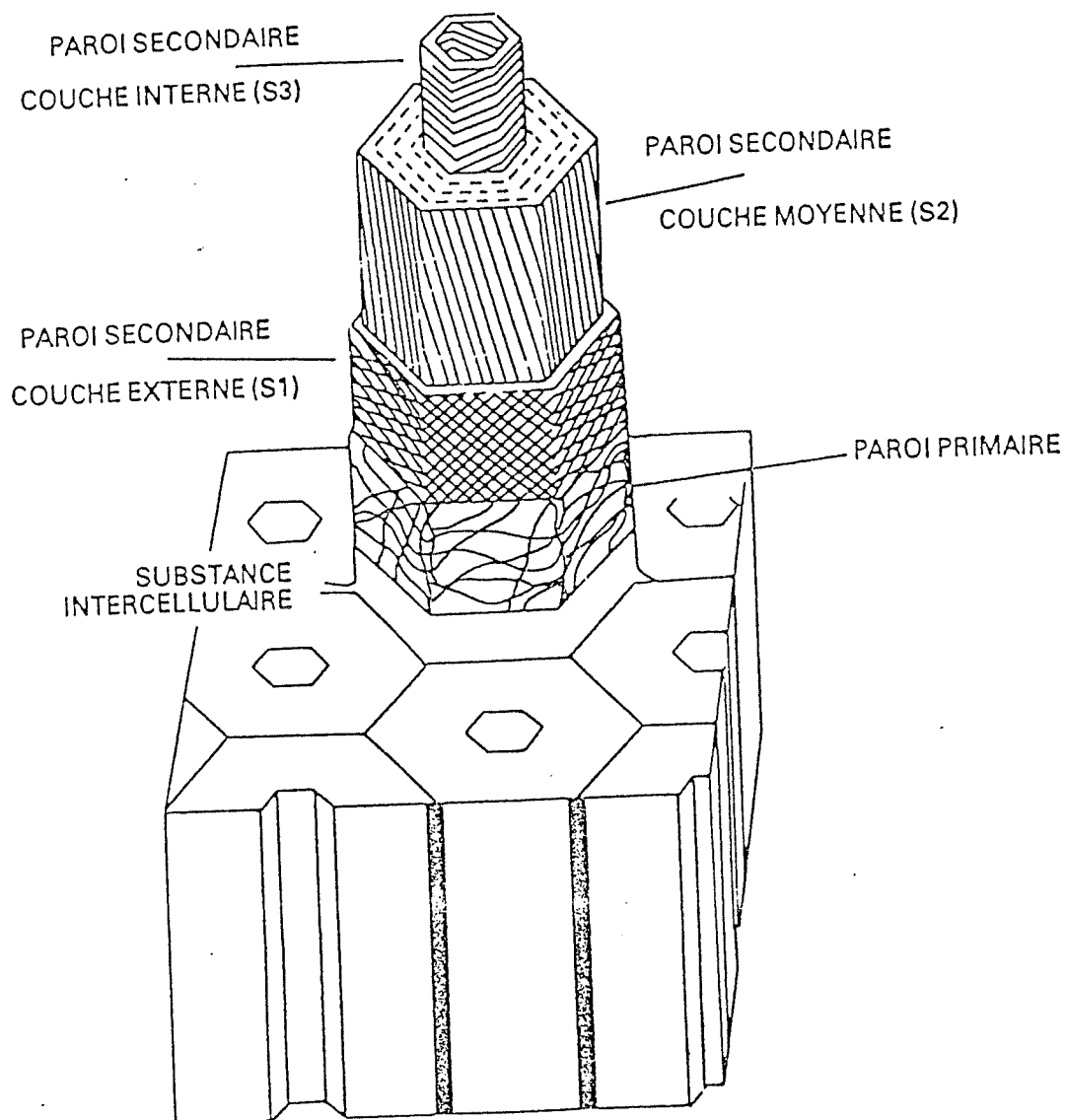


Figure 1.1: Schéma de la structure d'une fibre de bois.

lettre **P**), d'une paroi secondaire (paroi interne, désignée par la lettre **S**) et d'un canal au centre qu'on appelle le **lumen**. Dans le bois, les cellules sont cimentées entre elles par une substance appelée la lignine, ce qui donne à l'arbre sa rigidité structurale.

Les deux parois **S** et **P** sont constituées de cellulose en forme de microfibrilles, dont l'arrangement est différent suivant qu'ils sont en **S** ou en **P**.

La paroi primaire (**P**) est très mince et constituée d'une seule couche dans laquelle les microfibrilles sont espacées et disposées de façon désordonnée. Elle est généralement plus ou moins détériorée par le processus de défibrage. La paroi secondaire (**S**) est plus épaisse et forme la majeure partie de la fibre. Elle est composée de plusieurs lamelles. Dans chaque lamelle les microfibrilles sont disposées de façon parallèle et compacte (non espacée). Les microfibrilles de la lamelle ont la même orientation par rapport à l'axe de la cellule (orientation hélicoïdale centrée sur l'axe de la fibre). Sur la base de cette orientation on peut distinguer trois couches:

i) La couche externe S_1 . Elle est constituée de 4 à 6 lamelles orientées alternativement S et Z. S et Z sont les deux orientations hélicoïdales de sens opposés par rapport à l'axe de la fibre.

ii) La couche médiane S_2 . Elle constitue la partie principale de la fibre parce qu'elle est formée de 30 à 150 lamelles. Les microfibrilles de ces lamelles ont toutes la même orientation. L'angle d'orientation de ces microfibrilles définit les propriétés mécaniques de la fibre.

iii) La couche interne S_3 . Elle est formée de 0 à 6 lamelles de même orientation mais de sens opposé à S_2 .

La transition de S_1 à S_2 et de S_2 à S_3 se fait de façon progressive à travers un certain nombre de lamelles qu'on appelle lamelles de transition.

Il faut reconnaître qu'il existe des caractéristiques anatomiques de la fibre tels que (a) la longueur de la fibre, (b) l'épaisseur de la paroi de la cellule, (d) le diamètre du lumen, (c) l'angle d'orientation des microfibrilles de la couche S_2 par rapport à l'axe de la cellule... Par exemple, les fibres du bois du printemps (springwood fibres en anglais) se caractérisent par des parois minces, un grand lumen et un angle d'orientation des microfibrilles de S_2 petit. Les parois minces leur permettent de s'affaisser et de prendre la forme d'un ruban de 40 à 65 μm de largeur et de 10 à 12 μm d'épaisseur (deux fois l'épaisseur de la cellule) [1]. A l'opposé, les fibres du bois d'été (summerwood fibres) se caractérisent par des parois épaisses, un petit lumen et un grand angle d'orientation des microfibrille de S_2 . Les parois épaisses leur permettent de garder la forme cylindrique dont le diamètre varie entre 25 et 35 μm et une épaisseur de paroi qui varie entre 10 et 15 μm [1]. Ces propriétés anatomiques ont une influence directe sur les propriétés des fibres [2]. Les fibres du printemps se brisent facilement durant la mise en pâte et le raffinage, alors que les fibres d'été montrent une grande résistance et demeurent rigides, elles sont trois fois plus résistantes que les fibres du printemps. Ces caractéristiques ont des influences directes sur les propriétés mécaniques et rhéologiques du papier.

1.2.1.2. Nature physicochimique de la liaison interfibre.

La feuille de papier doit sa forme aux liaisons interfibrilles. A l'origine, ces liaisons ont été attribuées à l'interpénétration des microfibrilles et aux forces de friction entre les fibres. Swanson [3] a rapporté que cette thèse ne peut pas expliquer la résistance du papier. En effet, il n'y a qu'une faible différence de la résistance à la rupture entre une éprouvette sèche et une éprouvette imprégnée d'huile non polaire[4]. La liaison hydrogène ($-O-H \cdots O$) et les forces de van der Waals [3,5] ont été proposées pour expliquer le phénomène. Swanson a discuté l'implication des forces de van der Waals dans les systèmes celluloses et dans les liaisons interfibrilles.

Robertson [6] a comparé les courbes contrainte-déformation d'éprouvettes immergées dans différents liquides (figure 1.2). L'immersion dans l'eau fait perdre à l'éprouvette la quasi-totalité de ses propriétés mécaniques. Il a conclu par la suite que la contribution des liaisons hydrogène dans la liaison interfibre est la plus importante. Le fait que le papier réagisse de façon moins forte aux autres liquides, dont les molécules sont susceptibles de former la liaison hydrogène avec la cellulose, a été attribué à leurs tailles moléculaires. En effet, avec l'accroissement du volume molaire, les molécules du liquide deviennent plus encombrantes et moins réactives vis-à-vis des liaisons hydrogène de la liaison interfibre.

L'acétylation de la pâte de papier est la plus importante preuve de l'effet de la liaison hydrogène dans la liaison interfibre. La réaction consiste à remplacer les groupes

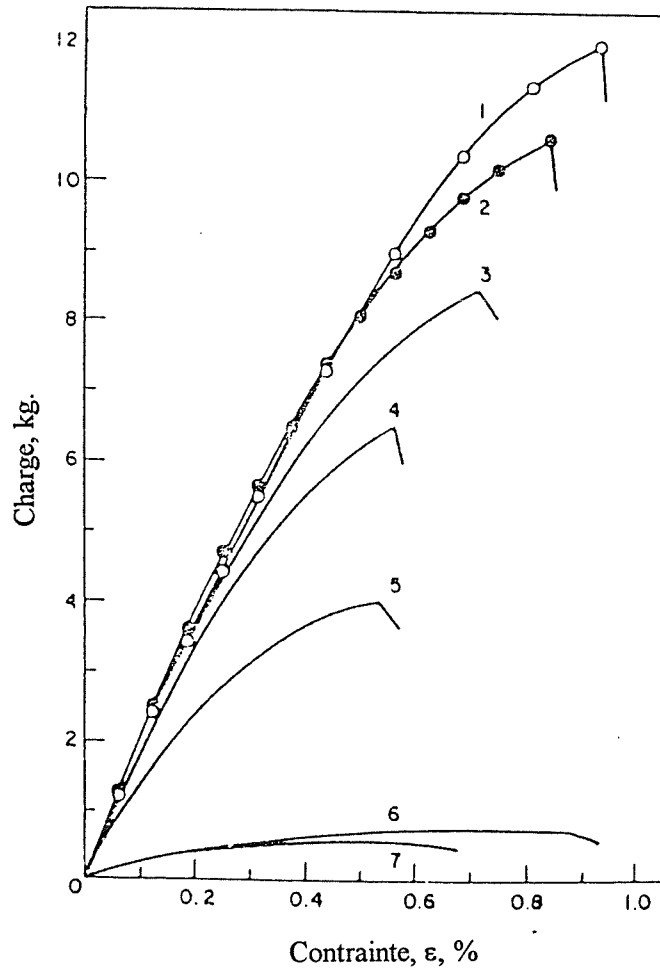


Figure 1.2: Courbes, traction-déformation, obtenues pour des éprouvettes de papier immergés dans différents liquides: (1) contrôle; (2) benzène; (3) vinyll acétate; (4) nitrométhane, (5) éthanol; (6) formamide; (7) eau, d'après ref. [6].

hydroxyls (-OH) par les groupes acétyls ($\text{CH}_3\text{-CO-}$). Ces derniers ne peuvent pas former de liaison hydrogène. Il en résulte que les propriétés mécaniques diminuent de façon drastique (Figure 1.3). McKenzie et Higgins [7] ont conclu à travers leurs expériences d'acétylation que seulement 1 sur 10^5 des groupes hydroxyls est impliqué dans la liaison interfibre.

En spectroscopie infrarouge, le groupe hydroxyl à l'état libre (qui ne présente pas la liaison hydrogène) est révélé par un pic d'absorption vers 3600 cm^{-1} . Ce dernier est généralement absent dans les matériaux cellulosiques comme illustré dans la figure 1.4. Il est très probable que les liaisons hydrogène sont impliquées dans des liaisons interfibre. Les courbes A et D montrent une forte absorption entre 3300 et 3500 cm^{-1} . La substitution des atomes d'hydrogène par des atomes de deutérium déplace l'absorption vers la région de 2500 à 2600 cm^{-1} , ce qui révèle un autre pic d'absorption entre 3200 et 3450 cm^{-1} . Ce pic a été attribué à la liaison hydrogène dans les deux cas: liaison interchaîne et liaison intrachaîne [8].

Pour comprendre les liaisons hydrogène impliquées dans la liaison interfibre, une partie de la chaîne de cellulose et sa configuration dans l'espace sont rapportées sur la figure 1.5. Les groupes hydroxyls sont de part et d'autre de l'axe de la chaîne et quelques-uns d'entre eux sont engagés dans des liaisons intramoléculaires. À l'état cristallin, les chaînes de cellulose sont parallèles et les groupes hydroxyls ne s'engagent en liaison intermoléculaire que si la distance qui sépare les deux atomes d'oxygène est comprise entre 2.4 à 3.2 \AA (Figure 1.6). Pour simplifier la représentation, la molécule est

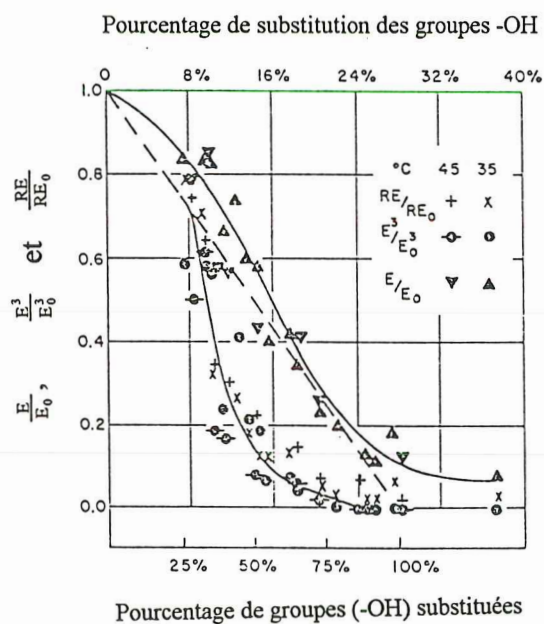


Figure 1.3: Détérioration de E (module de Young), E^3 et RE (énergie à la rupture), normalisé à leurs valeurs initiales, avec la substitution des groupes hydroxyles (-OH) par les groupes acétyles ($\text{CH}_3\text{-CO-}$). La réaction de substitution a été réalisée à deux températures (35 °C et 45 °C) [7].

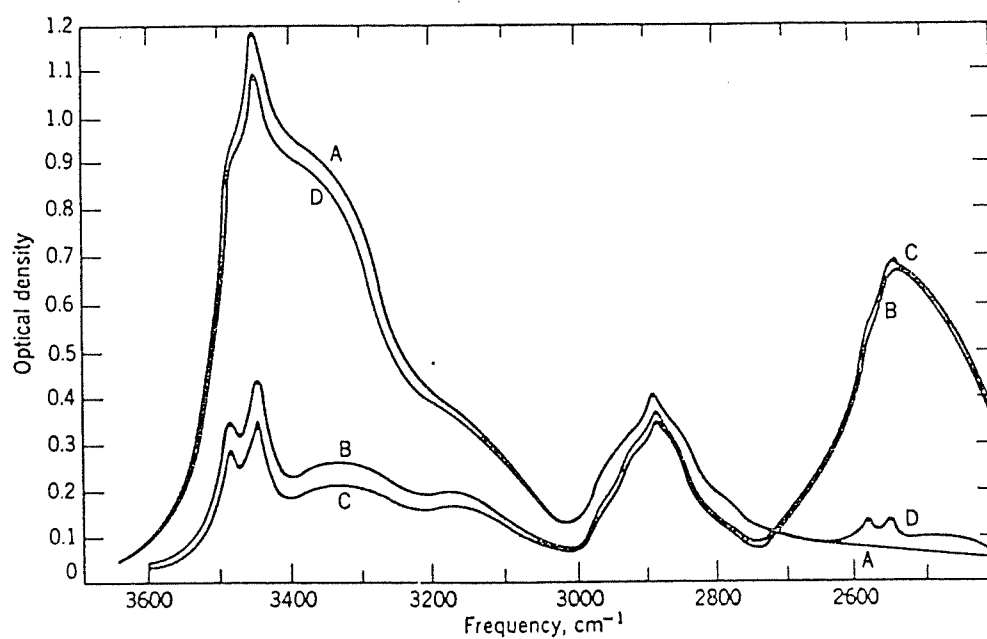


Figure 1.4: Spectre infra-rouge de la cellulose "viscose". (A) cellulose à l'état naturel; (B) substitution des atomes d'hydrogène par des atomes de deutérium dans la vapeur d'oxyde de deutérium; (C) substitution des atomes d'hydrogène par des atomes de deutérium dans le liquide d'oxyde de deutérium; (D) réhydrogénation dans l'eau [8].

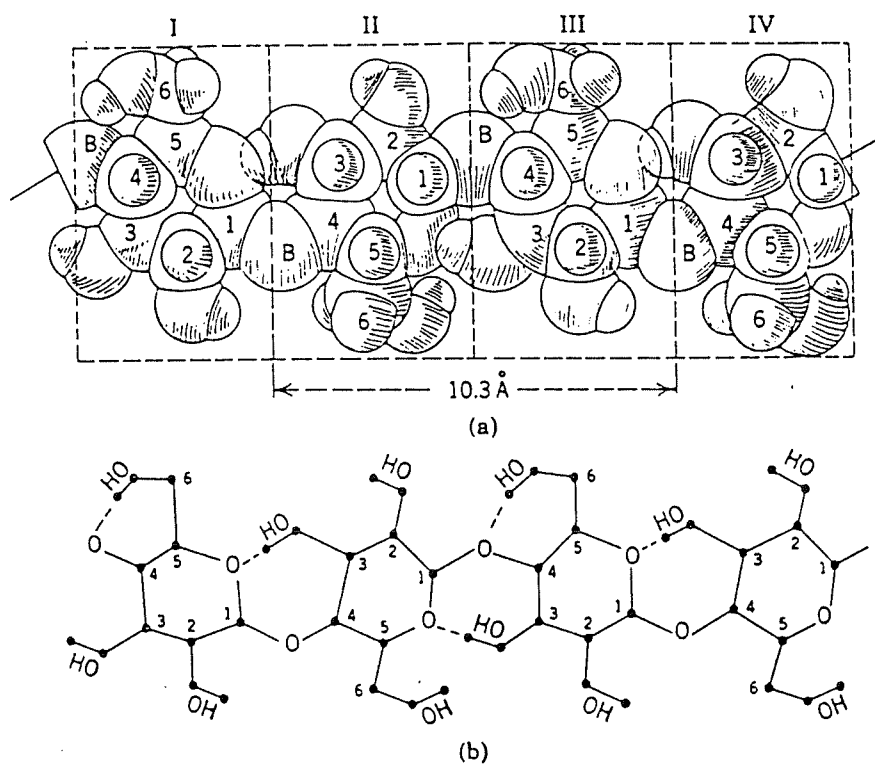


Figure 1.5: La molécule de cellulose et sa configuration dans l'espace. Quelques groupes -OH présentent la liaison hydrogène intramoléculaire [8].

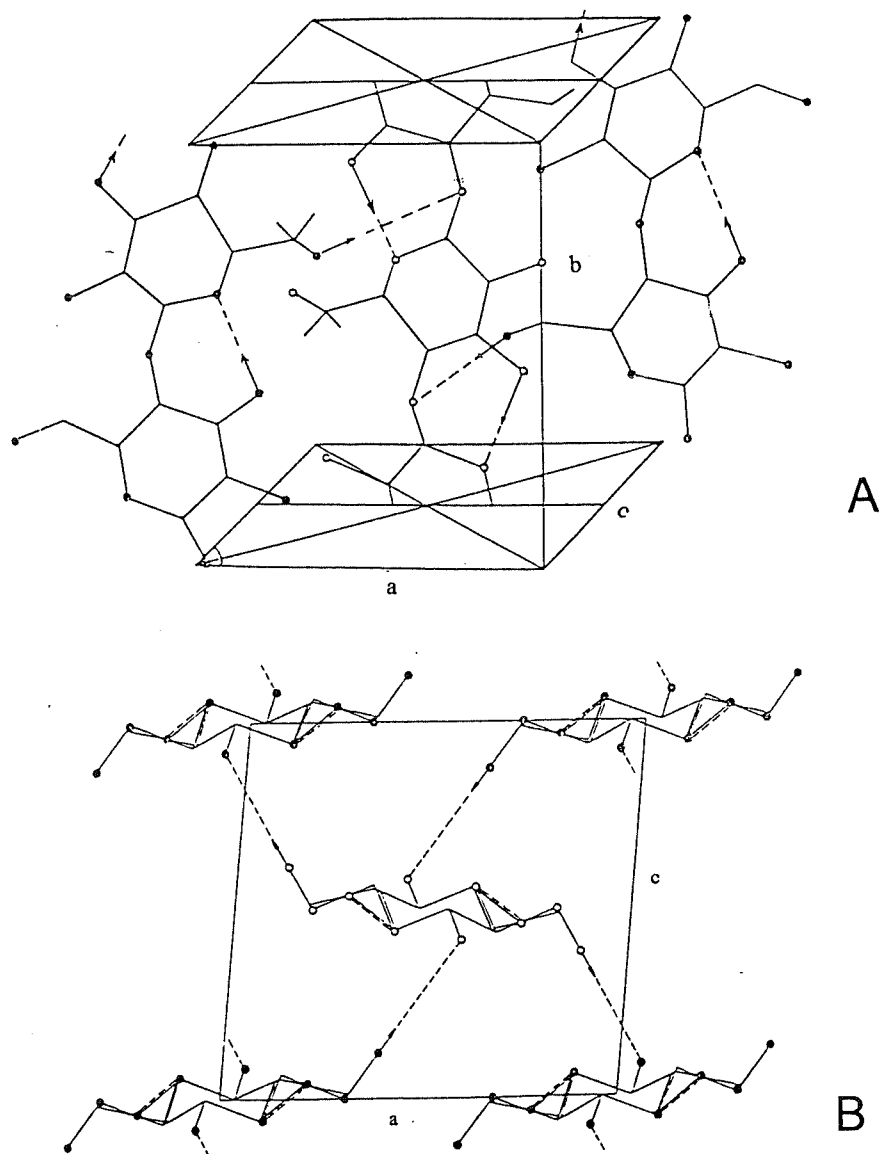


Figure 1.6: Disposition des chaînes de cellulose à l'état cristallin: (A) vue de côté; (B) vue à partir des extrémités des chaînes.

schématisée par un parallélépipède où les groupes hydroxyls sont sur deux faces opposées (parties hachurées) (Figure 1.7a) et l'arrangement des chaînes à l'état cristallin dans une microfibrille par l'empilement des parallélépipèdes (Figure 1.7b). L'intersection des zones hachurées des parallélépipèdes est le lieu de formation de liaison hydrogène.

1.2.1.3. Formation de la liaison fibre-fibre durant la formation et la consolidation du papier.

Avant le processus de formation de la feuille, les fibres sont mises en suspension dans l'eau à une consistance bien définie (généralement très faible). La suspension est ensuite drainée à travers une toile pour éliminer l'eau. On obtient un matelas de fibres qui contient 80% d'eau. À ce stade le contact entre les fibres se fait à travers des ponts de molécule d'eau (Figure 1.8b). Durant le séchage, la tension de surface promouvra, par élimination des molécules d'eau, certain points du contact au développement des liaisons hydrogène entre les surfaces des deux fibres (Figure 1.8c). A l'état sec, on distingue deux types de liaisons interfibres [9].

1) les liaisons directes; c'est-à-dire, les liaisons qui se développent aux croisements et aux recouvrements de deux fibres.

2) les liaisons indirectes; c'est-à-dire, le pontage des fibres par leurs fibrillations. A ce niveau, le lien moléculaire et l'interpénétration des macro- et microfibrilles contribuent simultanément à la liaison interfibre.

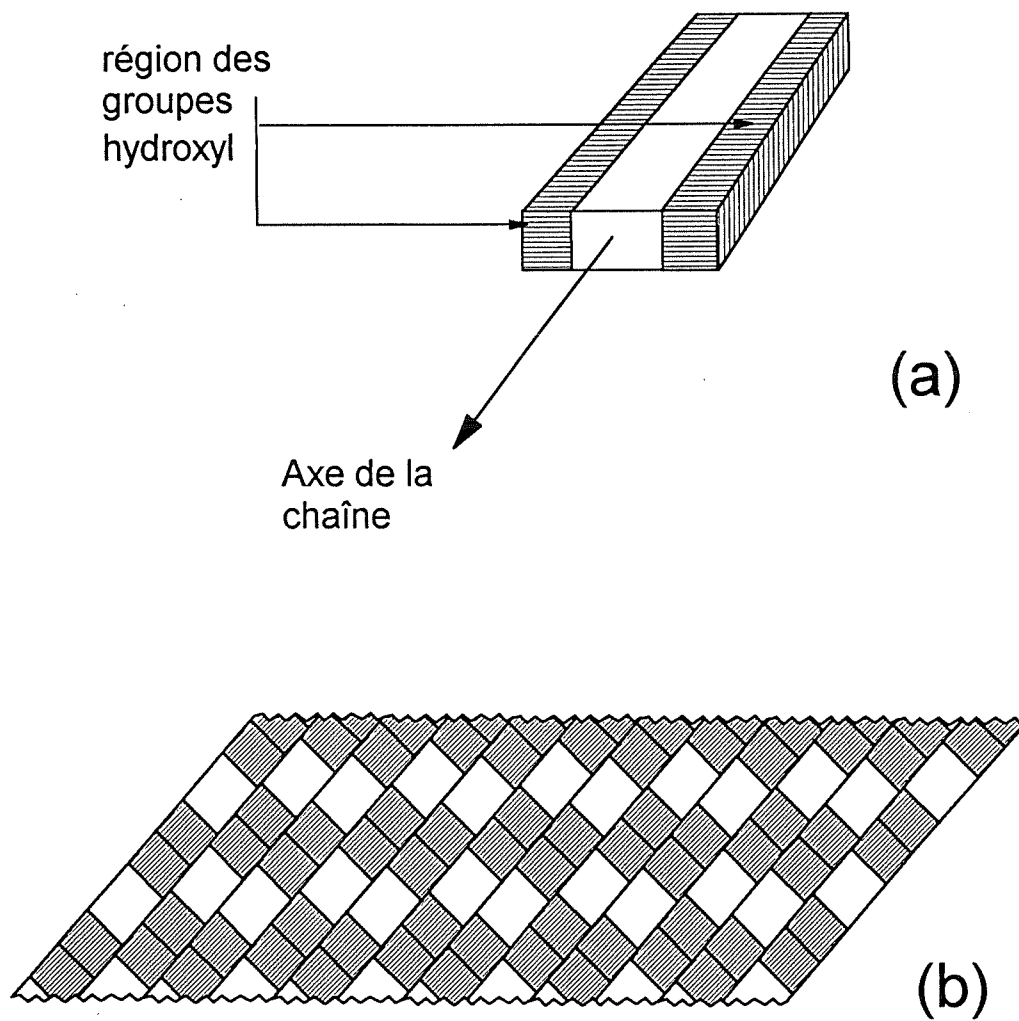


Figure 1.7: Schématisation de la chaîne de cellulose par un parallélépipède (a) et la microfibrille par l'empilement ordonné des parallélépipèdes (b).

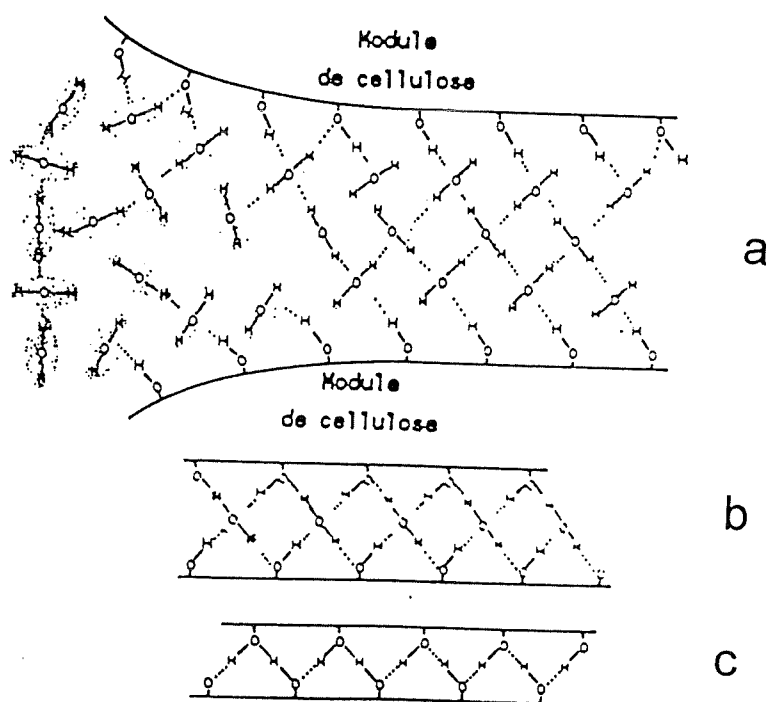


Figure 1.8: Schéma du processus de formation de la liaison interfibre à l'échelle moléculaire; a) fibre de cellulose en suspension dans l'eau, b) après drainage de l'eau, c) liaison interfibre dans une feuille sèche.

1.2.1.4. Mesures de la liaison interfibre.

Les facteurs les plus importants impliqués dans la liaison interfibre sont la surface de recouvrement entre les fibres et le nombre de points qui développent des liaisons dans l'aire de recouvrement. Différentes méthodes ont été soit développées ou adaptées à la mesure de la surface des liaisons interfibres.

1.2.1.4.1. Observation microscopique.

L'observation par microscope permet d'observer la forme et la taille de la surface de la liaison aux croisements et aux recouvrements des fibres. Page et coll. [10] ont utilisé le microscope optique avec un agrandissement moyen (x1060), sous une lumière polarisée pour observer la surface à l'état liée. La figure 1.9 montre un retraçage, à partir d'une photo, des limites des fibres et des surfaces de contact. Les surfaces en noir indiquent les zones qui ont développées la liaison interfibre. Il en découle que le croisement de deux fibres n'implique pas forcément le développement d'une liaison sur toute la surface de recouvrement, mais plutôt sur un nombre de points. Ils ont aussi rapporté que le nombre de points et la surface de la liaison sont influencés par la nature des fibres, le traitement mécanique qu'elles ont subi (raffinage) et le nombre de fibres impliquées au croisement (deux ou plus que deux).

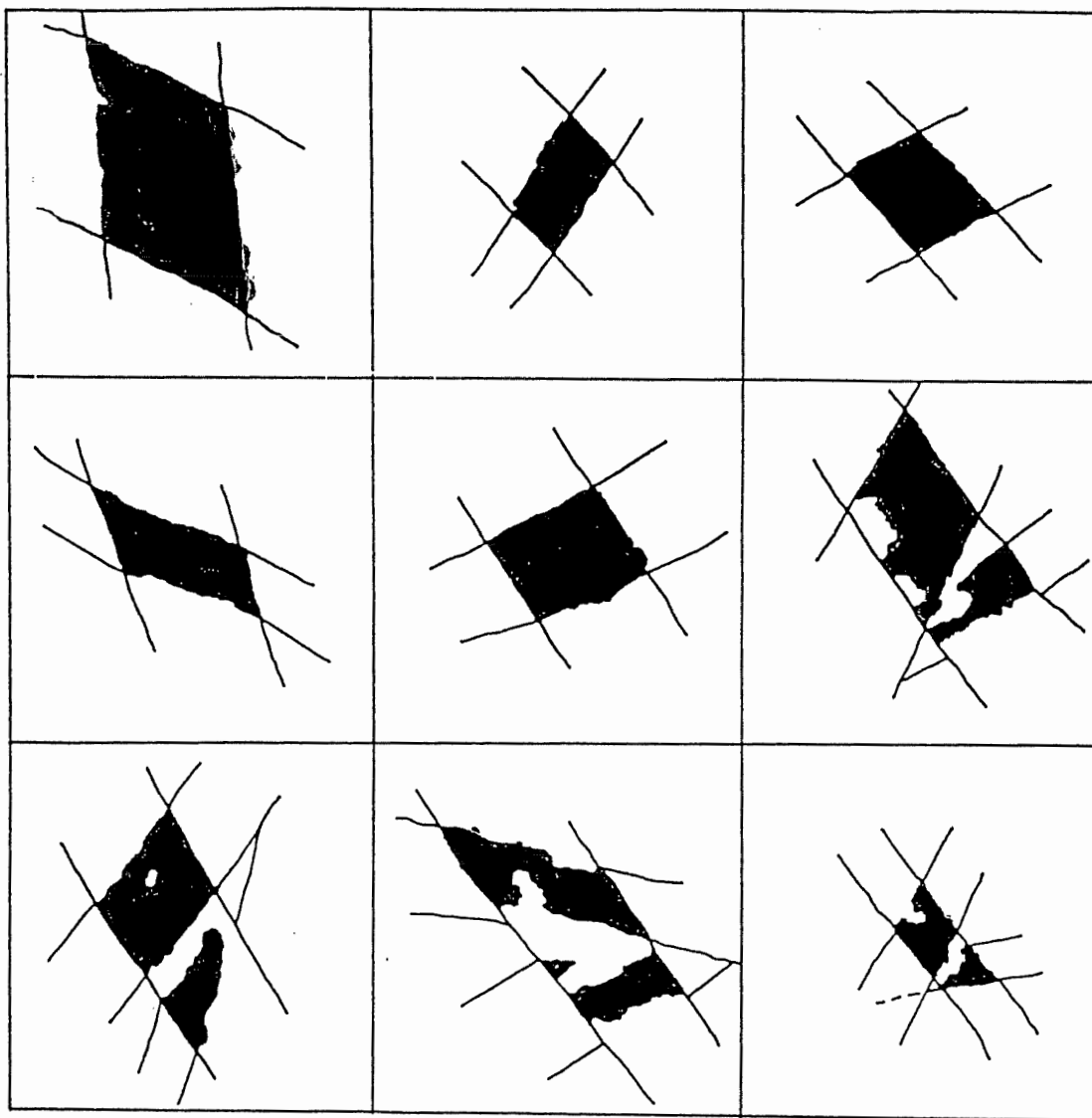


Figure 1.9: Retraçage des liaisons interfibres à partir d'une photo prise par le microscope optique [10].

Cette méthode a montré la complexité de la liaison fibre-fibre dans le papier. Cependant, il n'est pas toujours évident de retracer la forme des liaisons à partir des photos. En outre, les auteurs ont reconnu qu'il n'est pas toujours facile d'être objectif dans le choix des images et des zones d'échantillon qui reflètent les propriétés des liaisons à l'échelle du papier.

1.2.1.4.2. Méthode d'adsorption des gazs.

La technique d'adsorption d'azote est indirecte et mesure la surface totale des fibres à l'état non lié (sans liaison). Elle a été développée par Haselton [11,12] avec l'utilisation de l'équation de Brunauer, Emmett et Teller (BET). La technique consiste à faire passer à travers l'échantillon (préalablement séché) un mélange d'hélium-azote. En abaissant la température jusqu'à la température d'azote liquide, l'azote du mélange s'adsorbe à la surface des fibres ainsi exposée. Le volume de gaz adsorbé est proportionnel à la surface totale des fibres à l'état non lié.

La surface totale des fibres à l'état lié, dans un échantillon de papier, se détermine par la différence entre la surface totale des fibres, qu'on peut mesurer dans un amas de fibres (l'amas de fibres est un ensemble de fibres avec zéro liaison interfibre), et la surface à l'état non lié dans l'échantillon en question. L'amas de fibres utilisé pour mesurer la surface totale des fibres peut se réaliser de différentes façons, mais les méthodes les plus connues sont: «Méthode-1»: Changer l'eau de la suspension eau-fibre

par le benzène ou le n-butanol avant le séchage. «Méthode-2»: Sécher les fibres à partir d'un gel de fibre. «Méthode-3»: Sécher la suspension eau-fibre de faible consistance (0.2%) par étalement du mélange sur une surface de polyéthylène ou de Téflon [12,13]. Rennel [13] a rapporté que la surface totale des fibres dépend de la méthode de réalisation de l'amas. La comparaison des résultats avec les autres techniques de mesure de surface lui a permis de conclure que la surface mesurée sur l'amas réalisé par la «Méthode-3» est la plus proche de la réalité (Tableau I).

Tableau I. Comparaison des surfaces totales mesurées sur des amas préparés de trois façons différentes [13]

	Surface spécifique, $\text{m}^2 \cdot \text{g}^{-1}$.	
	Pâte de sulfite	Pâte de sulfate
Méthode-1	7.44	9.68
Méthode-2	2.33	2.59
Méthode-3	0.977	1.02
Extrapolation de la courbe « surface à l'état non lié » vs « résistance à la rupture (L) » à $L=0$ ^(a) .	0.602	0.715

^(a). C'est la détermination de la surface totale des fibres à partir de la relation entre la variation du coefficient de diffusion de la lumière et la résistance à la rupture. La méthode sera décrite au paragraphe suivant.

1.2.1.4.3. Méthode de diffusion de la lumière.

La diffusion de la lumière est la méthode indirecte la plus utilisée pour évaluer la surface à l'état lié. Elle est basée sur le fait que la diffusion est provoquée par le changement d'indice de diffusion à l'interface air-fibre. Il en résulte que seule la surface des fibres sans liaison diffuse la lumière. Le coefficient de diffusion S croît avec l'augmentation de la surface des fibres à l'état non lié. Il est plus important pour un amas de fibres que pour une feuille ordinaire ($S_{amas} > S_{feuille}$). Davis [14] a été le premier à suggérer la proportionnalité de S à la surface spécifique, A , (A est la surface par unité de masse à l'état non lié). Parsons [15], Ratiff [16], Kenney [17] et Leech [18] ont rapporté des résultats expérimentaux qui confirment cette linéarité. Cependant, leur méthode de détermination de la surface spécifique, A , a été controversée. Haselton [12] a utilisé la technique d'adsorption d'azote pour vérifier la proportionnalité de S à A . Il a trouvé une excellente linéarité entre S et la surface spécifique d'adsorption d'azote A_{BET} (figure 1.10)

$$S = k \cdot A_{BET} + i$$

L'ordonnée à l'origine " i " a été expliquée par le fait que même si toute la surface des fibres est à l'état lié ($A_{BET}=0$), la lumière diffusera par les deux surfaces de l'échantillon d'où $S=i < 0$. Swanson et coll. [19] ont trouvé que les valeurs de S , k et i dépendent de la nature des fibres et de la longueur d'onde utilisée. Par conséquent, le problème

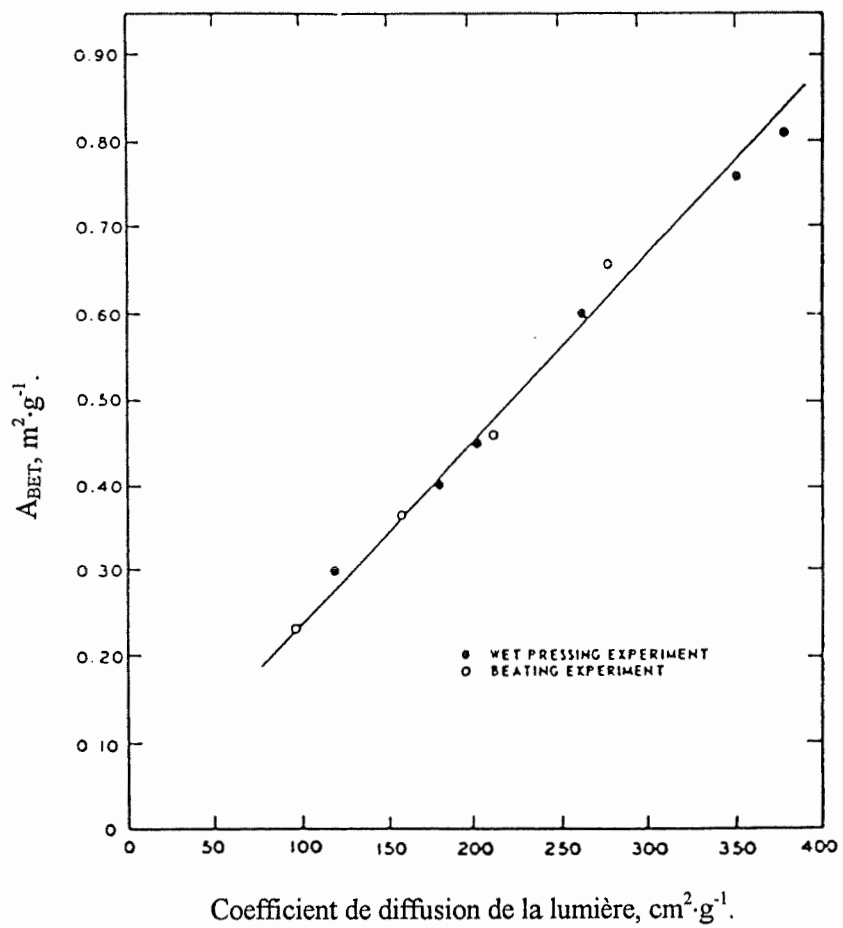


Figure 1.10: Variation du coefficient de diffusion de la lumière avec la surface à l'état non lié [12].

d'étalonnage de S se pose pour l'étude de chaque échantillon. Ratliff [16] a proposé une proportionnalité directe entre S et la surface par unité de masse à l'état non lié (A):

$$S = k'A$$

Pour décrire la signification physique de A, Ratliff [16] a discuté la différence entre la liaison effective (surface qui a développé une liaison à l'échelle moléculaire) et le contact optique en terme de distance séparant les deux fibres. Ces dernières forment la liaison hydrogène pour une distance qui ne dépasse guère une dizaine d'Angstrom (\AA), alors qu'elles sont en contact optique jusqu'à 150 \AA et même parfois jusqu'à 400 \AA dépendement de la longueur d'onde (Figure 1.11).

La surface à état non lié, A_u , et la surface totale, A_t , des fibres dans l'échantillon sont données respectivement par:

$$A_u = S_u/k' \quad \text{et} \quad A_t = S_t/k'.$$

La surface relative à l'état lié (RBA) est donnée par:

$$RBA = \frac{A_b}{A_t} = \frac{A_t - A_u}{A_t} = \frac{S_t - S_u}{S_t}.$$

Où A_b est la surface à l'état lié.

Les problèmes ne se sont pas arrêtés à ce point. En effet Haselton [12] et Rennel [13,20] ont rapporté que S_t dépend de la méthode utilisée pour réaliser l'amas de fibres et même du volume massique de l'amas lors de la mesure. Ingmanson et coll. [21] se sont basés sur l'idée qu'un échantillon sans liaison interfibre aura une résistance à la rupture nulle. Ils ont tracé S en fonction de la résistance maximale à la rupture (Fig. 1.12)

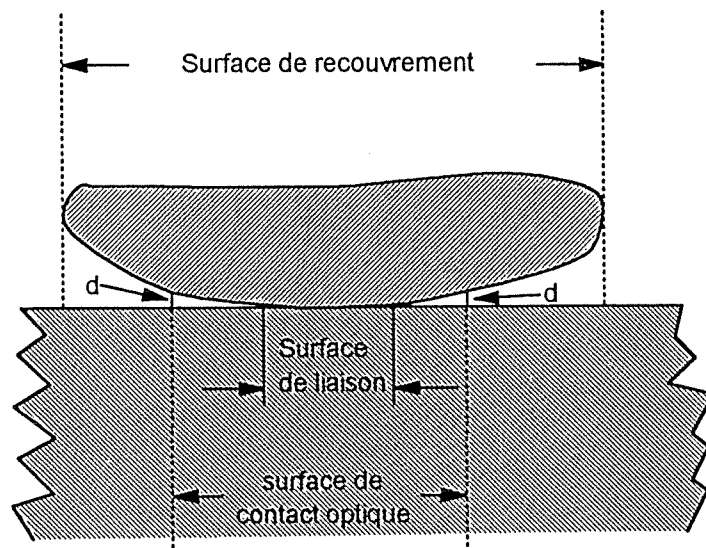


Figure 1.11: Liaison effective et contact optique entre deux fibres.

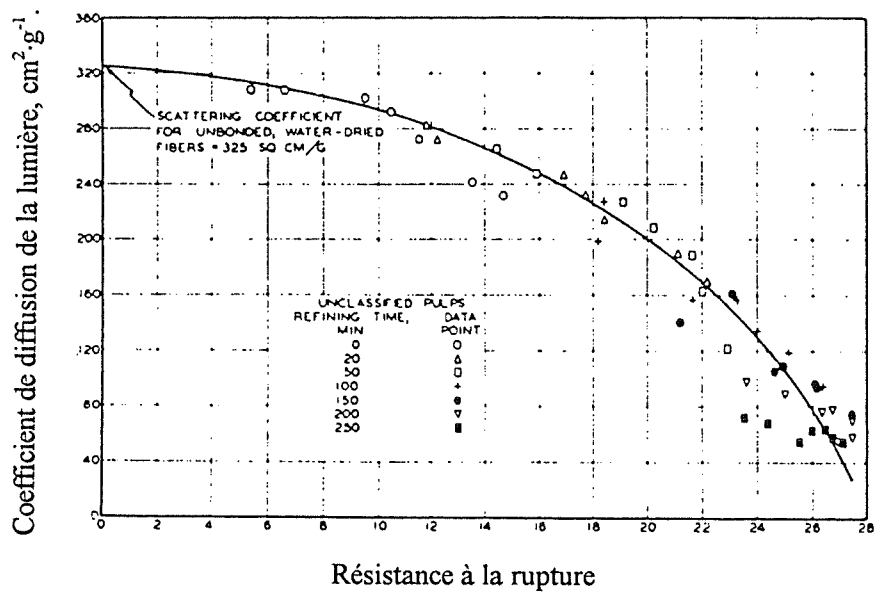


Figure 1.12: Relation entre le coefficient de diffusion de la lumière et la résistance à la rupture [21].

et ont extrapolé la courbe à $L=0$. La valeur S_t ainsi obtenue est présumée être la valeur correspondante à zéro liaison interfibre. Rennel [13] a rapporté que la conversion en surface du S_t obtenu par cette méthode est comparable à la valeur A_{BET} mesurée sur un amas séché directement à partir d'une solution eau-fibre (Tableau 1). Cependant, la méthode ne peut donner que S_t des fibres non raffinées.

1.2.2. Propriétés mécaniques du papier, des fibres et des liaisons interfibres.

Dans la feuille, les fibres sont aplaties en forme de ruban de dimension 0.01mm d'épaisseur, 0.02 mm de largeur et 1 mm de longueur. Elles forment un réseau à deux dimensions dont les connections (liaison interfibre) sont généralement parallèles au plan de la feuille. Il en résulte que les propriétés mécaniques dans le plan de la feuille sont généralement différentes de celles normales au plan. Quand une contrainte est appliquée dans le plan de la feuille, elle sera transmise d'une fibre à l'autre via les liaisons fibre-fibre. Les fibres seront généralement sollicitées dans leur longueur et les liaisons fibre-fibre seront soumises à des contraintes en cisaillement. Quand la contrainte est normale au plan, les fibres seront sollicitées dans leur épaisseur et les liaisons fibre-fibre subissent des contraintes en traction.

Dans ce qui suit, nous allons discuter des propriétés mécaniques des fibres et des liaisons interfibres et leurs contributions aux propriétés mécaniques de la feuille dans le plan et normale au plan, à l'instar des tests les plus utilisés.

1.2.2.1. Propriétés mécaniques dans le plan.

Le test de traction est le plus utilisé pour l'étude des propriétés mécaniques du papier dans le plan. Ceci est dû au fait qu'il n'y a que deux paramètres mis en jeu, à savoir, la contrainte et la déformation d'une seule direction. D'autres tests, dont la description théorique est plus complexe, ne sont utilisés que pour étudier un phénomène mécanique particulier.

1.2.2.1.1. Les différentes grandeurs mesurables et leurs unités dans le test en traction.

La courbe traction-déformation typique du papier est montrée à la figure 1.13.

Deux régions peuvent être distinguées:

1- Une région élastique qui s'étend de 0 à 0.3 ou 0.5% élongation et dans laquelle le papier se comporte comme un matériau Hookéen, c'est-à-dire que la contrainte varie linéairement avec la déformation.

2- Une région inélastique qui s'étend sur le reste de la courbe. Elle est caractérisée par une diminution graduelle de la pente qui finit parfois par se stabiliser à une certaine valeur et montrer une deuxième portion linéaire de la courbe. La longueur de la région inélastique dépend de l'humidité environnante et elle se caractérise par le développement de déformation permanente observée suite à des cycles traction-déformation.

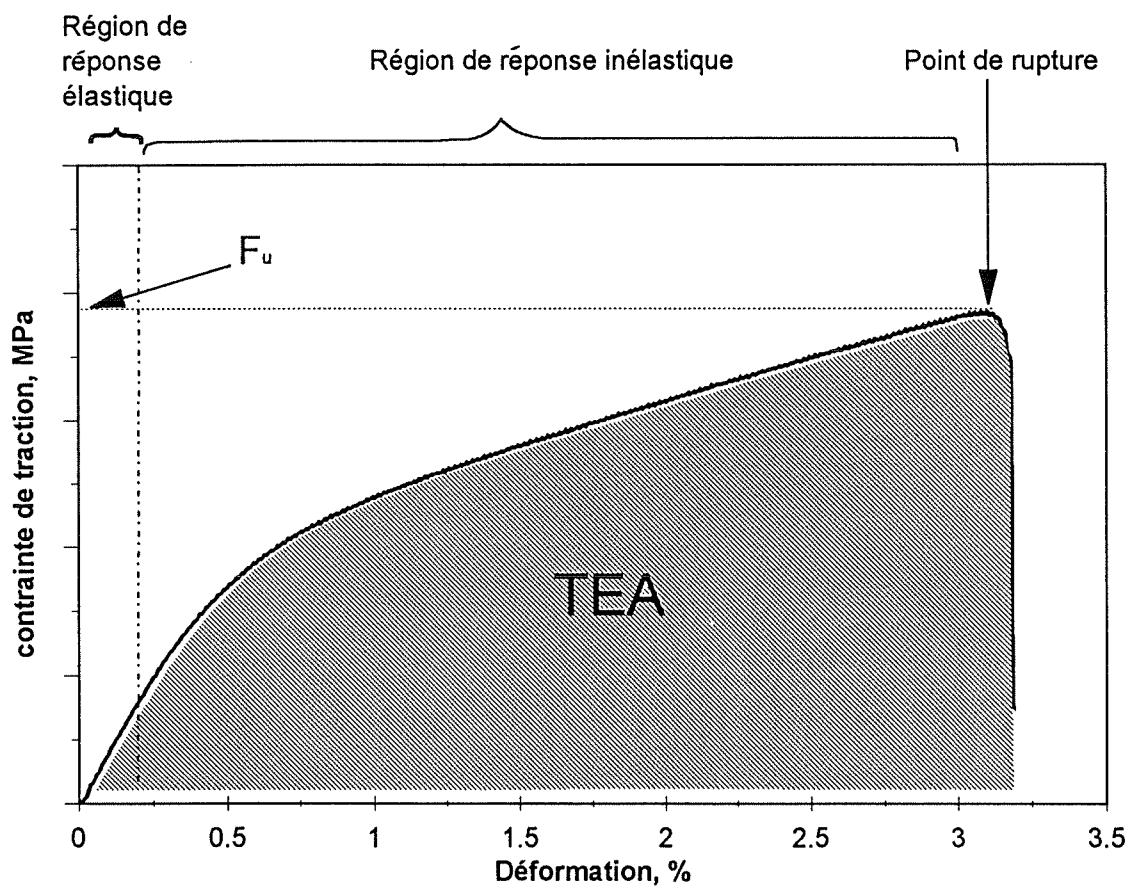


Figure 1.13: Courbe traction-déformation d'une éprouvette de papier.

Le papier montre aussi un comportement viscoélastique qui se manifeste par des réponses caractéristiques, tels que la dépendance de la courbe traction-déformation sur la vitesse de déformation, le fluage, la relaxation de la contrainte...

Les différentes grandeurs mesurables qui caractérisent l'échantillon étudié sont: le module de Young (E) (parfois appelé module d'élasticité, défini par la pente initiale de la courbe), la résistance maximale à la rupture (F_u), la déformation maximale et l'énergie de traction absorbée par l'échantillon (Tensile Energy Absorption TEA) (C'est l'énergie nécessaire à la rupture de l'échantillon divisée par la surface de la face de l'échantillon).

La résistance maximale à la rupture (F_u) est la force maximale à la rupture divisée par la largeur de l'échantillon. La contrainte maximale (σ_u) correspondante s'obtient en divisant F_u par l'épaisseur de l'éprouvette. Les unités de F_u , de σ_u et TEA dans les différents systèmes d'unité sont donnés dans le **tableau II**.

La définition conventionnelle de contrainte à la rupture se bute sur la vraie mesure de l'épaisseur. En effet, la mesure de l'épaisseur pour un matériau poreux, tel le papier, dépend de la pression utilisée par l'instrument sur l'échantillon. Pour s'affranchir des pores et déterminer une résistance à la rupture supportée juste par la partie solide de l'éprouvette, les papetiers ont choisi de substituer l'épaisseur par la densité de masse

Tableau II. Les unités conventionnelles de la résistance maximale à la rupture et la contrainte correspondante.

Grandeur	définition	Système International	Système cgs	Système d'unité anglais
F_u	Force par unité de largeur	$N \cdot m^{-1}$	dyne·cm ⁻¹ ou kgf·cm ⁻¹	lb·in ⁻¹
σ_u	Force par unité de surface	$N \cdot m^{-2}$	dyne·cm ⁻² ou kgf·cm ⁻²	lb·in ⁻²
TEA	Énergie par unité de surface	$J \cdot m^{-2}$	dyne·cm/cm ⁻² ou kgf·cm/cm ⁻²	lbf in/in ⁻²

surfaccique (m_A) et de définir la résistance à la rupture par la **longueur à la rupture, L**:

$$L = \frac{F_u}{g \cdot m_A}$$

Où g est l'accélération de la pesanteur. L'unité de L est le mètre au lieu des unités conventionnelles des forces (N) et des contraintes ($N \cdot m^{-2}$). Elle nous donne la résistance de la partie fibreuse de l'éprouvette. Le tableau III, illustre les différentes unités et la conversion de la contrainte spécifique maximale σ_u^* (défini par $\sigma_u^* = F_u/m_A$) en longueur de rupture L .

Tableau III. Conversion de la contrainte spécifique en longueur de cassure

Système d'unité	contrainte spécifique $\sigma_u^* = F_u \cdot m_A^{-1}$				÷	Accélération g	=	Longueur de cassure	
	Valeur	Unité	or	valeur				unité ^a	valeur
S.I.	1	$\frac{\text{Nm}}{\text{kg}}$	or	1	$\frac{(\text{kg m} \cdot \text{s}^{-2})\text{m}}{\text{kg}}$	9.8	$\text{m} \cdot \text{s}^{-2}$	0.102	m
cgs	1	$\frac{\text{dyn} \cdot \text{cm}}{\text{g}}$	or	1	$\frac{(\text{g cm} \cdot \text{s}^{-2}) \cdot \text{cm}}{\text{g}}$	980	$\text{cm} \cdot \text{s}^{-2}$	$1.02 \cdot 10^{-3}$	cm
	1	$\frac{\text{kgf} \cdot \text{cm}}{\text{kg}}$	or	980	$\frac{(\text{kg cm} \cdot \text{s}^{-2}) \cdot \text{cm}}{\text{kg}}$	980	$\text{cm} \cdot \text{s}^{-2}$	1	cm
Anglais	1	$\frac{\text{lbf} \cdot \text{in}}{\text{lb}}$	or	32.2	$\frac{(\text{lb in} \cdot \text{s}^{-2}) \cdot \text{in}}{\text{lb}}$	32.2	$\text{ft} \cdot \text{s}^{-2}$	1	in

^a 1N est équivalent à 1 kg x 1 m·s⁻²; 1 dyne est équivalent à 1 g x 1 cm·s⁻²; 1 kgf est équivalent à 1 kg x 980 cm·s⁻²;

1 lbf est équivalent à 1 lb x 32 ft·s⁻²

1.2.2.1.2 Propriétés mécaniques des fibres et des liaisons interfibre.

La réponse des fibres et des liaisons interfibres à la sollicitation en traction d'une éprouvette de papier a fait l'objet de plusieurs travaux. Généralement, les travaux publiés visent la réponse dans une des trois régions de la courbe contrainte-déformation: à savoir, la partie élastique, la partie inélastique et le point de rupture.

A- Région élastique.

Le module de Young caractérise le comportement élastique du papier. Il est fonction de la nature de la pâte et des traitements qu'elle a subis. Il varie entre 300 et 600 km pour la pâte mécanique et entre 1000 et 1200 km pour la pâte chimique [22]. (Nous notons que E est généralement exprimé en unité de longueur comme pour la résistance à la rupture.)

Cox [23] a attribué le module de Young à la réponse des fibres et a développé une théorie pour décrire E. Il a considéré le papier comme un réseau à deux dimensions avec une orientation aléatoire des fibres et une distribution uniforme de la contrainte. Sa théorie a abouti au fait que le module de Young du papier est équivalent au tiers (1/3) de celui de la fibre. Page et coll. [22,24 ,25] ont démontré que les facteurs qui contrôlent E sont le module d'élasticité des fibres, le degré de liaison dans la feuille et la présence de boucles, de noeuds, d'ondulations et de microcompressions.

B- Région inélastique

La partie inélastique de la courbe a été décrite au début par Nordman et coll. [26 - 29] comme étant dû au processus progressif de cassure des liaisons interfibrilles. Ils ont montré que l'énergie, W , dissipée durant les cycles de déformation en traction varie linéairement avec la variation du coefficient de diffusion de la lumière ΔS (figure 1.14). Ils ont expliqué la variation de ΔS par la création de nouvelles surfaces par cassure des liaisons interfibrilles. Ils ont défini la ténacité de la liaison interfibrille (bond strength), appelée par la suite «Nordman bonding strength», par la pente de la courbe W en fonction de ΔS . Plusieurs critiques ont été publiées sur la signification physique du «Nordman bond strength». Van den Akker [30] a indiqué la grande disparité entre l'estimation théorique de la ténacité de la liaison interfibrille (de l'ordre de 10^{-2} à 10^{-1} J·m⁻²) et l'énergie de Nordman (23 J·m⁻²) une fois le coefficient de diffusion converti en surface. Uesaka [31] a rappelé que pour un matériau ductile tel que le papier, qui contient des défauts et des fissures, la majeure partie de l'énergie est dissipée sous forme de déformation plastique aux alentours des défauts.

C- Point de rupture.

Le point de rupture est caractérisé par la résistance maximale à la rupture de l'éprouvette. Cette grandeur est généralement utilisée par les papetiers à titre de facteur

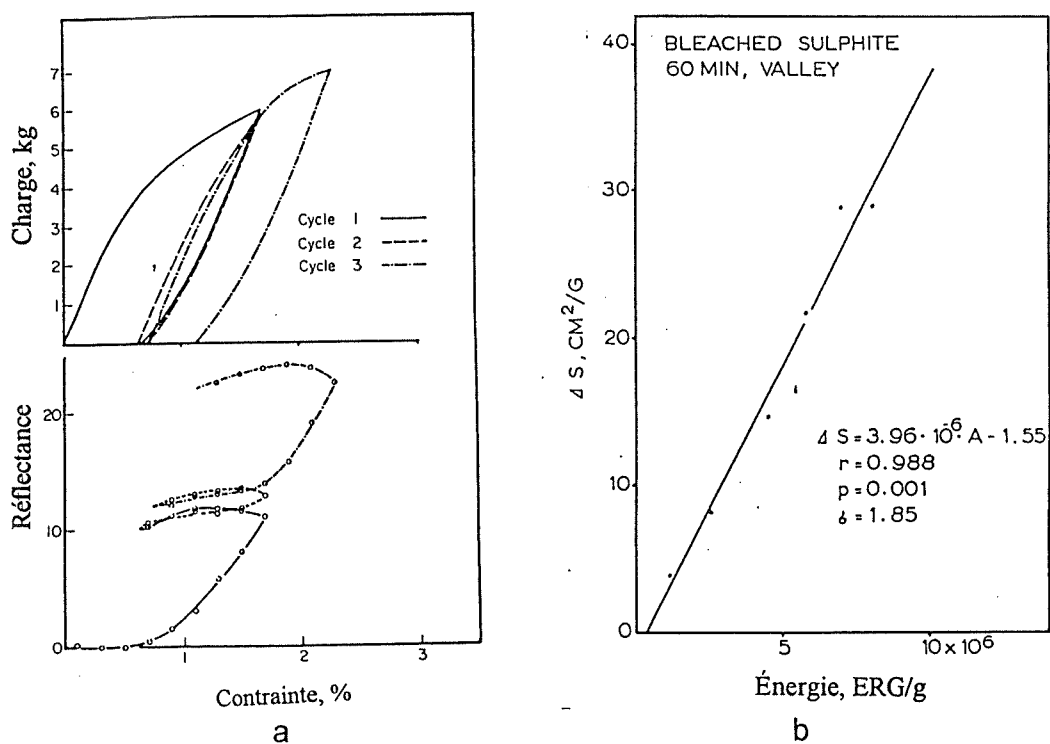


Figure 1.14: (a) variation cyclique de la traction et de la réflectance avec la déformation dans le papier. (b) variation du coefficient de diffusion de la lumière ΔS (déterminée à partir de la réflectance) avec l'énergie dissipée durant les cycles de déformation [28].

de contrôle de qualité. Dinwoodie [32], dans une revue, a rapporté l'influence de la morphologie des fibres, telles que la longueur et la densité, sur les propriétés du papier et spécialement sur la résistance à la rupture. Van den Akker et coll. [4] ont étudié la ligne de rupture sous le microscope et ont déterminé le pourcentage des fibres cassées et les fibres tirées. Le pourcentage élevé de fibres cassées (entre 55% et 65%) les a conduit à conclure que la liaison interfibre est plus résistante que prévue. Le pourcentage de fibres cassées augmente avec le raffinage (de 40 à 70%) et la pression humide (de 53 à 60%). Il croît aussi avec les additifs qui fortifient la liaison interfibre (de 56 à 75%) et diminue avec les additifs qui affaiblissent la liaison interfibre (de 56 à 11%). Kayama et coll. [33] ont conclu aussi que la résistance à la rupture des liaisons est plus importante que la résistance à la rupture des fibres. Helle [34 -36] a montré qu'il y a d'autres facteurs qui gouvernent le pourcentage de fibres cassées à la ligne de rupture après avoir analysé différents papiers. Il a formulé les observations suivantes:

a) Le pourcentage de fibres cassées est plus élevé pour les fibres sulfite que pour les fibres kraft dans des feuilles ayant la même résistance à la rupture. (Sulfite et Kraft sont deux méthodes chimiques de défibrage du bois.)

b) Le pourcentage de fibres cassées est plus élevé pour la pâte à haut rendement que pour la pâte à faible rendement pour la même résistance à la rupture. (Le rendement est le rapport de la masse fibre obtenu sur la masse initiale de bois.)

c) Le pourcentage des fibres cassées augmente avec la vitesse de déformation.

d) Le pourcentage de fibres cassées est plus élevé pour une feuille séchée sous contrainte standard que pour une feuille séchée sans contrainte, bien que la première montre une surface relative de l'état lié plus faible (RBA).

Il a conclu dans un de ses articles [34] que la résistance à la rupture d'une fibre prise individuellement, est différente de sa résistance dans le papier. Il a aussi suggéré que les liaisons interfibres sont plus sollicitées dans une éprouvette à faible degré de liaison que dans une éprouvette à grand degré de liaisons interfibres. Dans le dernier cas, la probabilité d'initiation de la rupture par concentration de la contrainte est plus importante.

Kallmes et coll. [37 -40] ont proposé une théorie pour le comportement du papier en traction-déformation. Ils ont postulé à l'existence d'une RBA critique au-dessous de laquelle l'endommagement sera initié par la rupture des liaisons et au-dessus de laquelle, l'endommagement sera initié par la cassure des fibres.

Page [41] a développé un modèle semi-empirique où il a exprimé la longueur de rupture (résistance à la rupture) du papier (T) en fonction de la résistance à la rupture des fibres, décrite par la résistance d'une éprouvette à longueur nulle (Z), la surface relative à l'état lié (RBA), la section moyenne (A), le périmètre moyen (P) et la longueur moyenne des fibres (L), la résistance à la rupture en cisaillement par unité de surface de la liaison (b), la densité de la matière fibreuse (ρ), et l'accélération de la pesanteur (g).

$$1/T = 9/8Z + 12A\rho g/bPL(RBA)$$

Il a montré que cette équation montre un excellent accord avec les résultats publiés par Van Den Akker et coll. [4] et Helle [34] concernant la variation de T avec le taux de fibres cassées, et un bon accord de la linéarité de la fonction $1/T$ ($1/(RBA)$) et les résultats de Ingmanson [21]. Cette équation a été utilisée dans le travail de Page et dans d'autres travaux, par la suite, pour déterminer la résistance à la rupture (b) de la liaison interfibre.

Cette équation montre que la résistance à la rupture est fonction des propriétés mécaniques (résistance à la rupture) et morphologique (longueur et section) des fibres, et des propriétés des liaisons interfibres (degré de liaison et résistance à la rupture). Mais ne tient pas compte des déformations plastiques dues aux redressements des noeuds, courbures et microcompressions existantes dans les fibres

1.2.2.2. Propriétés du papier dans l'épaisseur

Puisque la plupart des surfaces à l'état lié sont parallèles au plan de la feuille, le test de traction dans l'épaisseur, c'est-à-dire normale au plan, devrait refléter les propriétés mécaniques des liaisons fibre-fibre. Deux méthodes ont été développées pour cet objectif, à savoir, le test de traction normale au plan, le «z-direction tensile test», et le test de pelage « Peel test ».

1.2.2.2.1. Test de traction normale au plan.

Le test de traction normale au plan consiste à coller chaque face de l'éprouvette à une face carrée, de 1 pouce de côté, d'un bloc métallique par un adhésif de forte résistance de rupture. L'ensemble composé des deux blocs métalliques et l'éprouvette au milieu est monté dans un appareil de traction tel que l'Instron (Figure 1.15). La traction est appliquée dans le but de séparer les deux blocs.

La courbe traction-déformation est rapportée sur la figure 1.16. Elle ne présente pas de région linéaire reproductible et caractéristique du papier étudié, c'est-à-dire, une région qui reflète les propriétés élastiques du papier dans l'épaisseur. Van Liew [42] a rapporté que l'épaisseur sous contrainte varie à cause de la pénétration de l'adhésif dans les pores de l'éprouvette. Il en résulte que la contrainte et la déformation sont locales et nonuniformes dans l'échantillon. L'échantillon commence à s'endommager dès l'application de la contrainte jusqu'à la rupture. Wink et coll. [43] ont rapporté que la résistance à la rupture et l'énergie nécessaire à la rupture de l'éprouvette diminuent avec l'accroissement de la densité de masse surfacique. Ce résultat a été attribué à la prépondérance de la concentration de la contrainte dans le test. Il en découle la difficulté d'utiliser la méthode pour déterminer la ténacité et la résistance à la rupture de la liaison interfibre.

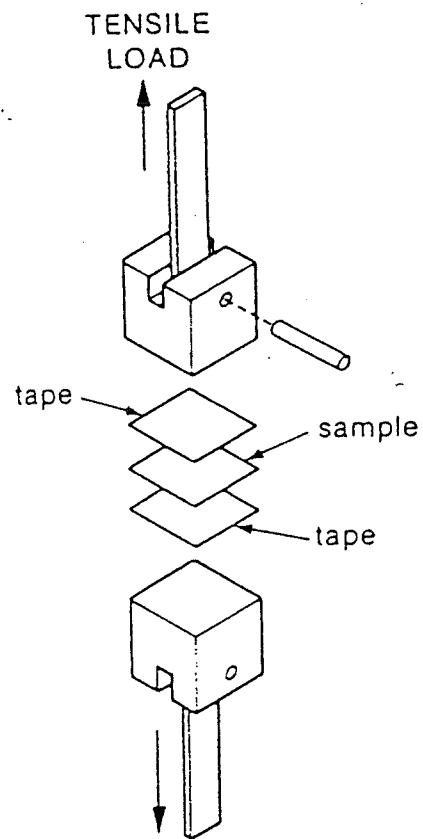


Figure 1.15: Description de test de traction en épaisseur.

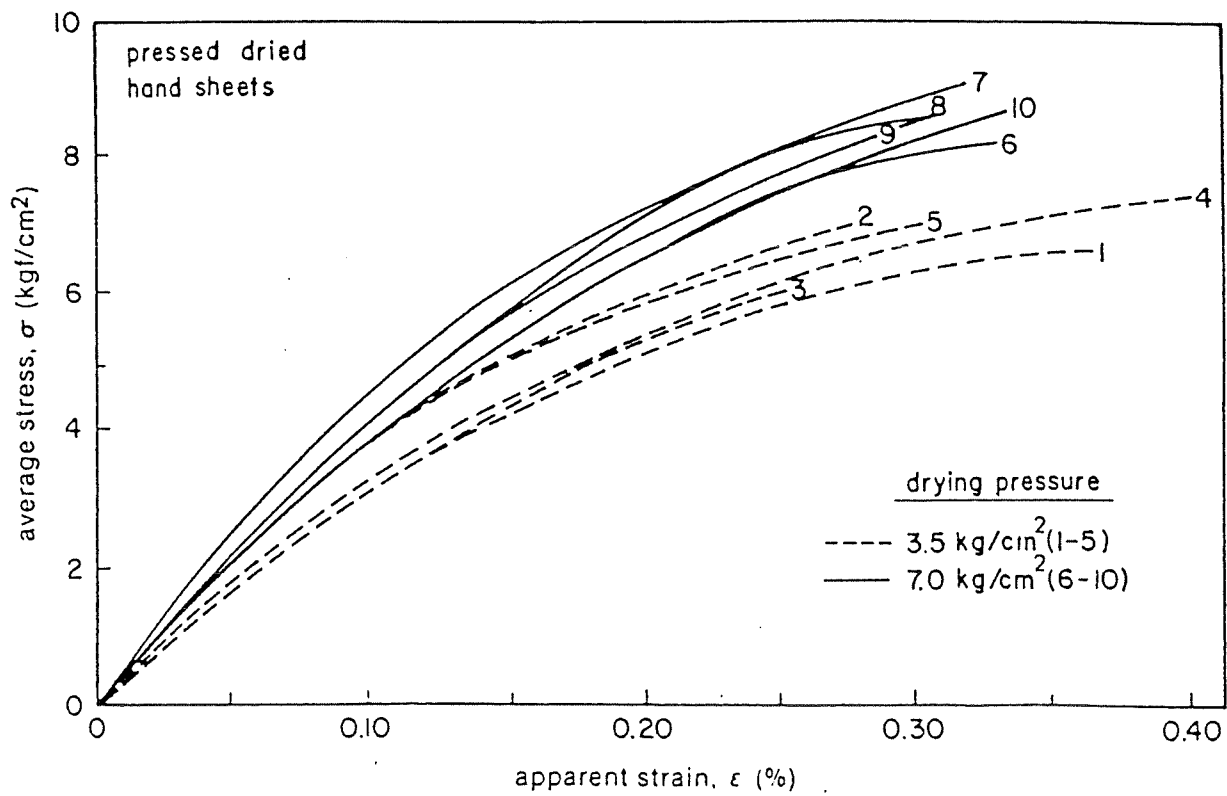
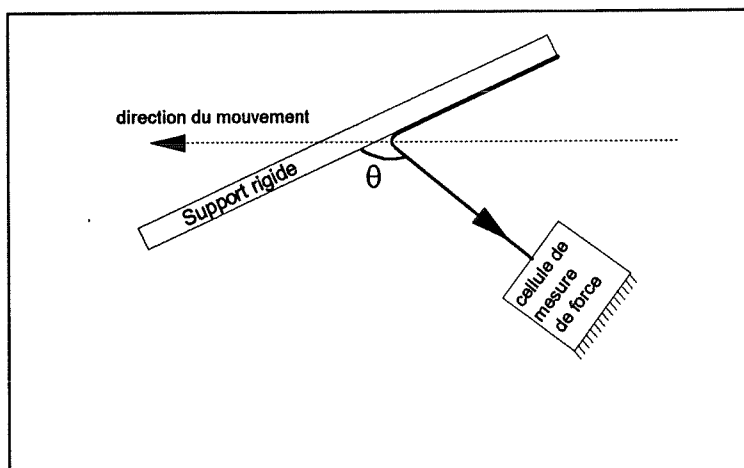
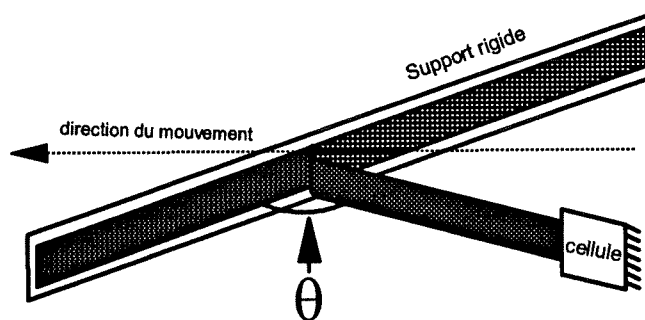


Figure 1.16: Courbe traction-déformation en épaisseur pour des échantillons identiques réalisés aux pressions humides 3.5 Kg/cm² (courbes de 1 à 5) et 7.0 kg/cm² (courbes 6-10) [42].



a



b

Figure 1.17: Schématisation du test de pelage avec un des adhérents rigides
(a) vue de face; (b) vue de profile.

1.2.2.2.2. Le test de pelage.

Le test de pelage consiste à détacher un adhérent flexible d'un substrat rigide ou flexible (Figure 1.17). Il se caractérise par sa simplicité en manipulation et la simplicité de configurer et d'adapter toute machine de traction pour ce genre d'expérience. Durant le pelage, l'appareil mesure la force de pelage instantanée (P) qui est reliée à l'élément de travail de pelage (dW) par la relation $dW = P(1 - \cos\theta)dl$, où θ est l'angle de délaminage et dl l'élément adhérent détaché. L'énergie de pelage par unité de surface est donnée par:

$$W_p = \frac{dW}{b \cdot dl} = \frac{P}{b}(1 - \cos\theta).$$

Où b est la largeur de l'adhérent détaché.

Cette méthode a été introduite récemment dans le domaine du papier par Skowronski et Bichard [44] afin de déterminer la ténacité de la liaison interfibre dans la direction z . L'expérience consiste à coller, aux deux faces d'un échantillon rectangulaire, deux rubans adhésifs transparents de même dimension. Les rubans adhésifs servent à renforcer les deux couches ainsi séparées. L'angle de délaminage a été fixé à 90° et W_p a été considéré comme une mesure de l'énergie de rupture de l'échantillon (ténacité de l'échantillon dans l'épaisseur). Le très faible taux de fibres cassées observé après le processus de délaminage les a conduits à penser que toute l'énergie dissipée a servi à la création de nouvelles surfaces. Ils ont défini la «ténacité spécifique de la liaison, SBS» par

le rapport de W_p à la variation du coefficient de diffusion de la lumière (ΔS), $\frac{W_p}{\Delta S}$.

$\Delta S = S_{av} - S_{ap}$. S_{av} et S_{ap} sont respectivement les mesures du coefficient de diffusion avant et après délaminage. Cependant, la méthode ne peut pas mesurer la résistance à la rupture de la liaison, autrement dit la contrainte maximale à la rupture dans la direction de l'épaisseur.

Skowronski et Bichard [44] ont rapporté que W_p croît avec l'augmentation du degré de liaison qui s'opère par le raffinage ou la pression humide. Cependant, la ténacité de la liaison, SBS, est pratiquement constante. Stratton [45] a montré que W_p augmente avec l'ajout de fines (débris) et d'autres substances chimiques connues pour leur effet d'accroître la ténacité de la liaison interfibre. Il a rapporté que l'accroissement de W_p , avec ces additifs, ait été plus important que l'accroissement du module d'élasticité mesuré par ultrason dans la direction normale au plan. Il a conclu que le test de pelage est une méthode sensible aux propriétés de la liaison fibre-fibre normale au plan.

Retulainen et Ebeling [46] et Dasgupta [47] ont rapporté la variation du SBS avec l'accroissement de la surface à l'état lié (RBA) par raffinage. Contrairement au résultat de Skowronski, ils ont trouvé une diminution de SBS avec l'accroissement du raffinage. Retulainen et coll. [46] ont spéculé sur le fait que le raffinage réduit la rigidité du papier en flexion, ce qui peut être une des causes de la réduction du SBS.

Skowronski [48] a aussi rapporté que W_p et SBS croissent avec la vitesse de délaminage. Yamaushi et coll. [49] ont rapporté la variation de la force (implicitement le travail, W_p) avec la vitesse et l'angle de pelage. Cependant, ils ont observé que l'accroissement, de la force avec la vitesse, s'accompagne du changement du mode de rupture d'interfacial, à faible vitesse, à cohésive (dans le papier) à grande vitesse.

1.2.3. Conclusion.

Les propriétés mécaniques du papier dans le plan de la feuille ont été largement étudiées. La contribution des fibres et des liaisons interfibres a été mise en évidence durant le test de traction. Différents modèles ont été développés pour décrire la réponse des fibres et des liaisons interfibres et même de quantifier la résistance à la rupture de la liaison. Cependant, les propriétés du papier dans l'épaisseur ont été moins étudiées. Ceci est dû au fait que les problèmes techniques liés à la variation de l'épaisseur dans le test de traction sont difficiles et même impossibles à éliminer. En plus, il est très difficile de mesurer l'accroissement de la surface totale des fibres due à la cassure des liaisons interfibres. Par conséquent, cette méthode ne peut pas être utilisée pour mesurer la ténacité et la résistance à la rupture.

Dans l'application du test de pelage, le problème de la mesure de l'accroissement de la surface totale par cassure des liaisons interfibres ne se pose pas. Cependant, le fait

d'attribuer toute l'énergie de pelage à la cassure des liaisons interfibres est basé seulement sur une supposition non confirmée (pelage élastique). De plus, d'autres questions restent posées, comme:

1- Comment peut-on expliquer l'effet de l'angle et de la vitesse?

2- Quel est l'effet rhéologique des rubans adhésifs utilisés pour renforcer les parties détachées?

3- Comment expliquer le fait que la ténacité diminue avec le raffinage?

Ces questions ont été l'objet de ce travail. En effet, notre but de départ a été d'arriver à élucider l'effet des paramètres de pelage (vitesse et angle de pelage) et les paramètres reliés à l'échantillon (épaisseur, raffinage et pression humide) sur la ténacité de la liaison interfibre.

Il nous est apparu utile de faire une revue générale des modèles proposés pour décrire le pelage et les circonstances de leurs applicabilités, de même, s'il existe un modèle qui pourrait répondre à la nature du papier.

1.3. Le pelage

Le pelage est un des moyens les plus utilisés pour l'étude des adhésifs. Industriellement, de tels tests sont souvent employés pour caractériser les adhésifs sensibles à la pression, et en particulier les rubans adhésifs qui sont difficiles à tester par d'autres méthodes. La configuration du test de pelage, le plus utilisé, est schématisé sur la figure 1.17. Ce test consiste à avoir au moins un adhérent flexible collé à un substrat rigide ou flexible et solidement lié à un support rigide. L'angle peut prendre toutes les valeurs de zéro à 180°. Cependant, les angles 90° et 180° sont les plus utilisés.

Les tests de pelage se divisent en deux groupes: « pelage élastique » et « pelage inélastique ». Si l'adhérent à délaminer fléchit élastiquement et que la propagation de la flexion se fait sans dissipation d'énergie, le **pelage est élastique**. Dans le cas contraire, la flexion se fait de façon inélastique et la propagation de la flexion nécessite de l'énergie, c'est un **pelage inélastique**.

1.3.1. Pelage élastique.

Les premiers modèles développés ont été destinés à la description du pelage élastique. En effet, dans ce cas toute l'énergie sert à la rupture dans la zone de séparation, ce qui

facilite sa description mathématique. Deux approches ont été proposées: l'une, basée sur un bilan énergétique, permet de calculer le taux de restitution de l'énergie; l'autre, axée sur l'analyse de la forme de la zone de détachement, permet l'analyse des constantes dans la zone de rupture.

1.3.1.1. Bilan d'énergie.

Supposons qu'un adhérent inextensible et de largeur b , soumis à la force P à l'angle θ , est pelé sur une longueur dl (Figure 1.18). Le travail de pelage élémentaire est donné par: $d\Omega = P \cdot dX$,

où dX est le déplacement subi par la force. dX est relié à la distance délaminée dl par:

$$dl \cdot (1 - \cos\theta) = dX$$

L'élément du travail sera donné par:

$$d\Omega = P \cdot dl \cdot (1 - \cos\theta)$$

Si l'on divise $d\Omega$ par l'élément de surface délaminée ($b \cdot dl$), nous obtenons une énergie par unité de surface.

$$W = \frac{d\Omega}{b \cdot dl} = \frac{P}{b} (1 - \cos\theta)$$

W est l'énergie d'adhérence à l'interface. Elle a été considérée indépendante de l'angle θ .

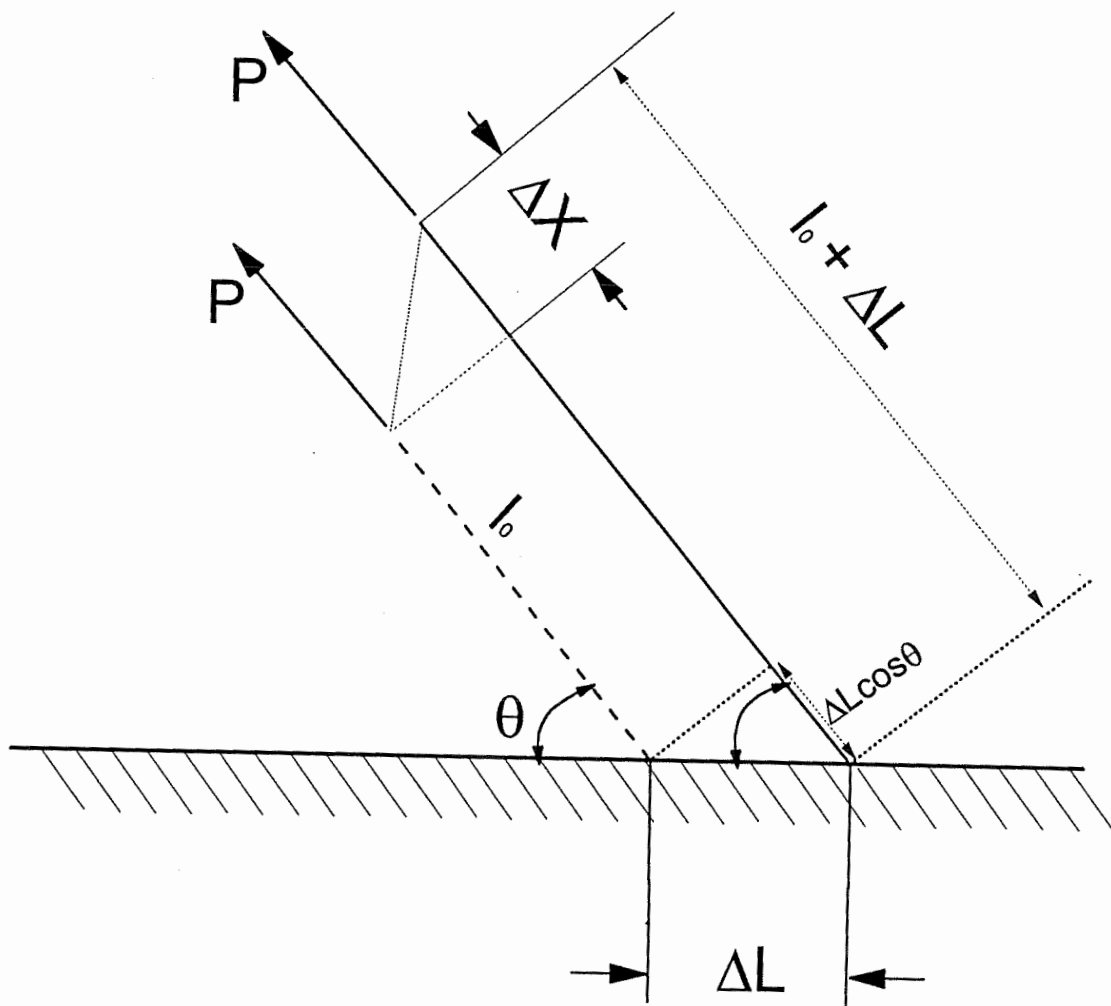


Figure 1.18: Schéma décrivant la relation entre le déplacement de la force (ΔX) et la portion délaminée (ΔL).

L'inconvénient majeur de cette approche est qu'elle ne rend pas compte du profil de la bande pelée, ni de l'effet et de la nature des contraintes présentes à l'interface.

1.3.1.2. Forme de la zone de détachement.

Spies [50] et Bikerman [51] ont analysé la situation d'un adhérent flexible collé à un substrat rigide par l'intermédiaire d'un adhésif d'épaisseur h_0 . Ils ont négligé les contraintes de cisaillement et ont utilisé la théorie des faibles flexions pour développer les équations qui gouvernent la variation de la contrainte et de la déformation de l'adhésif dans la zone de détachement:

$$M = \frac{EI}{R} = EI \frac{\frac{d^2y}{dx^2}}{\left(1 + \left(\frac{dy}{dx}\right)^2\right)^{1.5}} \approx EI \frac{d^2y}{dx^2}$$

Où M est le moment de flexion, EI est la rigidité à la flexion et R le rayon de courbure.

Pour simplifier le problème, ils ont aussi supposé que l'adhérent et l'adhésif sont de nature hookéenne dans la zone de détachement. L'expression qu'ils ont obtenue pour la force (F_m) de pelage à 90° est:

$$F_m = 0.3799 \cdot b \cdot \sigma_0 \left(\frac{E}{E_a}\right)^{0.25} \delta^{0.75} h_0^{0.25}$$

Où b est la largeur de la bande à délaminer, σ_0 est la résistance à la rupture de l'adhésif, E est le module de l'adhérent, E_a est le module de l'adhésif, h_0 est l'épaisseur de l'adhésif, et δ est l'épaisseur de l'adhérent.

Cette expression montre la dépendance de la force à la largeur de la bande et aux différents paramètres de l'adhérent et de l'adhésif. Cependant, la force calculée pour un adhésif qui présente le comportement le plus proche du comportement hookéen, est de 3 à 6 fois plus grande que celle mesurée [52].

En se basant sur la même théorie des faibles flexions, Kaelble [53] a développé un des modèles les plus compréhensibles pour le délaminage élastique. Il a considéré que la zone de détachement est sujette à des contraintes en cisaillement et en traction (nommée parfois contrainte de clivage) (figure 1.19). Il a développé les expressions de ces forces (cisaillement et traction) séparément, puis, les a combinées pour estimer la force de pelage à tout angle. Les contraintes de traction (σ) et de cisaillement (λ) ainsi obtenues sont fonction de la distance x du point de rupture ($x=0$) et sont données par:

$$\sigma = \sigma_0 e^{\beta x} (\cos \beta x + k \sin \beta x)$$

$$\lambda = \lambda_0 e^{\alpha x}$$

$$\text{avec } \beta = \frac{E_a \cdot b}{4E \cdot I \cdot h_0}, \quad k = \frac{\beta \cdot m}{\beta \cdot m + \sin \theta} \quad \text{et} \quad \alpha = \frac{G}{\sqrt{E \cdot h_0 \cdot \delta}}$$

où I est le moment d'inertie de l'adhérent, m est le moment fléchissant, et λ_0 est la résistance à la rupture en cisaillement.

Les courbes montrant les variations des contraintes σ et λ se trouvent à la figure 1.20. La contrainte en cisaillement décroît exponentiellement à partir du point de rupture. La contrainte en traction montre une distribution harmonique dans la zone non

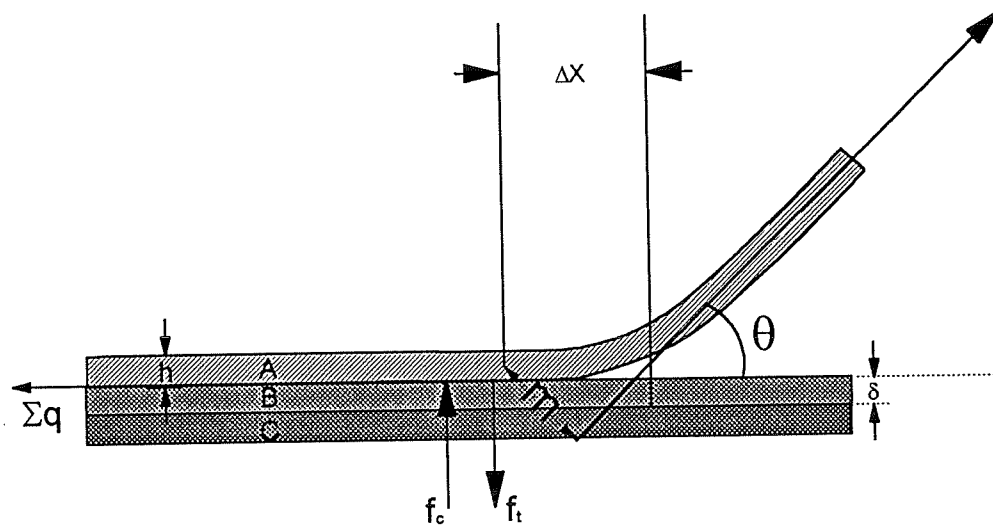


Figure 1.19: Schéma des différentes forces (clivage « f_c, f_t » et cisaillement « Σq ») appliquées sur l'adhérent durant le délaminage [53].

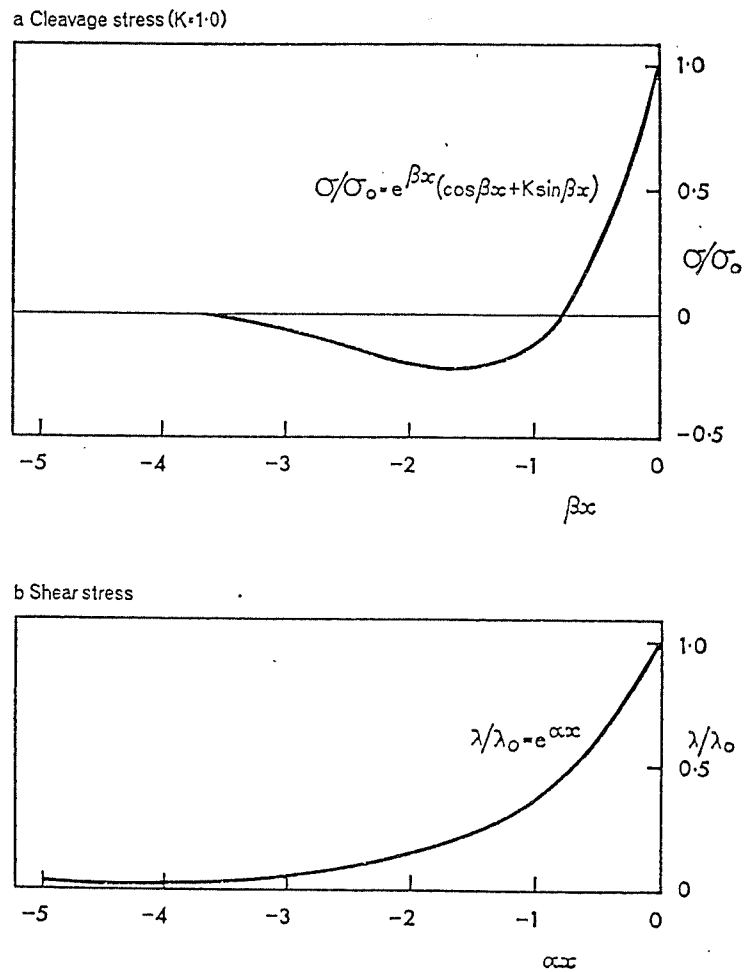


Figure 1.20: Distribution théorique des contraintes de clivage normalisée à la valeur initiale (a) et de cisaillement normalisées à la valeur initiale (b) dans la zone détachement en fonction de la position à partir du point de rupture [53].

pelée, c'est-à-dire, une succession de zones de compression et de traction dont l'amplitude diminue très rapidement (Figure 1.20 a).

Kaelble a combiné ces contraintes pour déterminer l'expression de la force de pelage en fonction de l'angle θ :

$$P = bh_0 \left[\frac{k \cdot \sigma_0}{\sqrt{2E_a}} + \frac{\sqrt{3} \cdot \lambda_0 \cdot \cos\theta}{\sqrt{2G}} \right]^2 \cdot (1 - \cos\theta)^{-1}$$

Si $90^\circ \leq \theta \leq 180^\circ$, les contraintes en cisaillement seront négligeables et l'expression de P sera réduite à:

$$P = \frac{bh_0 k^2 \cdot \sigma_0^2}{2E_a \cdot (1 - \cos\theta)}$$

Cette théorie ne s'accorde que qualitativement avec les résultats expérimentaux. En effet, elle ne prévoit pas l'effet de la vitesse sur la force de pelage tel qu'il est montré par les résultats expérimentaux (figure 1.21). De même, les résultats ne montrent pas que la force P est inversement proportionnelle à $(1 - \cos\theta)$ pour les grands angles. Cependant, la présence du minimum entre 30° et 40° (aux vitesses 0.02 et $0.2 \text{ in}\cdot\text{min}^{-1}$) a été attribuée au passage du mode de rupture par cisaillement au mode de rupture par clivage.

L'effet de la vitesse a été attribué par Yamamoto et Hayashi [54] au comportement viscoélastique de l'adhésif. Ils ont utilisé une méthode matricielle pour décrire la variation du comportement de l'adhésif avec la vitesse.

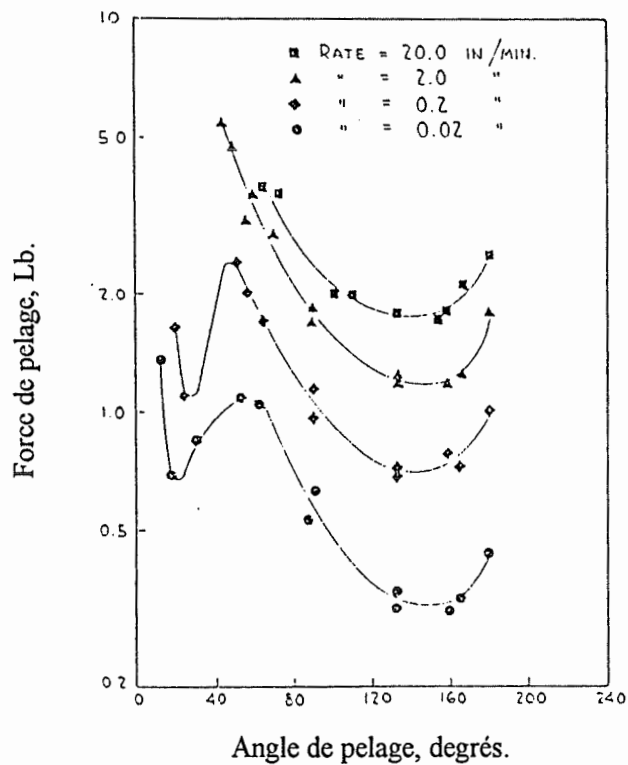


Figure 1.21: Résultats expérimentaux de la variation de la force de pelage avec l'angle et la vitesse de délaminage pour un ruban adhésif de cellophane [53].

1.3.2. Pelage inélastique.

Gent et Hamed [55] ont décelé l'existence de contradictions dans la théorie de Kealble [53], à savoir, le désaccord entre l'approximation des faibles flexions ($dy/dx \ll 1$) et les valeurs que k doit prendre pour les grands angles (spécialement pour 180°). Par conséquent, ils ont conclu que les suppositions de base, telles que la flexion faible et élastique et le comportement Hookéen des différents constituants, sont très restrictives. Suite à l'observation de courbures résiduelles après délaminage, ils ont plutôt suggéré l'existence de déformations inélastiques dans l'adhérent. Ultérieurement, ils ont développé [56] un modèle basé sur la théorie élémentaire de flexion pour calculer la force nécessaire à la propagation de la flexion à 180° dans les matériaux de comportement élastique-plastique:

$$P_y = \frac{\sigma_y e_y t}{4} \left[\frac{t}{2R e_y} + \frac{2R e_y}{t} - 2 \right],$$

Où σ_y et e_y sont respectivement la contrainte et la déformation à la limite d'élasticité, R est le rayon de courbure, et t est l'épaisseur de la couche à fléchir.

Ils ont aussi suggéré que les comportements «pelage élastique» et «pelage inélastique» dépendent de l'épaisseur. En effet, pour des adhérents très minces ou très épais, l'énergie dissipée en flexion est très faible et même négligeable devant l'énergie de détachement (énergie nécessaire à la séparation à l'interface), d'où le comportement de pelage élastique. Cependant, pour les valeurs intermédiaires c'est le pelage inélastique qui

prévaut. Plusieurs travaux ont suivi et ont montré l'existence de déformation inélastique sur des matériaux présentant le comportement elasto-plastique [57, 58].

Aravas et coll. [59] ont rapporté que la propagation de la flexion dans les matériaux élastique-plastique peut se présenter sous la forme d'un cycle moment-courbure $M(\rho)$ (Figure 1.22). En approximant les différentes parties de la courbe moment-courbure par des fonctions $M(\rho)$, ils ont déterminé l'expression de l'énergie dissipée en flexion en fonction de la courbure. Mantel et Descaves [60] ont repris la même méthode pour décrire le pelage en «T». Ils ont supposé que les courbes traction-déformation et compression-déformation sont identiques. Utilisant trois des modèles les plus connus pour les matériaux élastique-plastique, ils ont tracé les cycles moment-courbure correspondants. Ces résultats ont servi pour décrire le pelage d'un sandwich métal-polymère-métal.

Pour décrire le comportement viscoélastique de l'adhérent et l'influence de la vitesse, Kendall [61] a utilisé la théorie des faibles flexions pour décrire la forme de l'adhérent élastique durant le pelage. Il a ensuite substitué le module de Young, $E =$ constante (comportement élastique), par une fonction du temps $E=f(t)$ (comportement viscoélastique). Kim et Aravas [62] ont développé une formulation générale du problème pour un film qui présente une viscoélasticité linéaire. Leur résultat est une équation integro-différentielle dont la résolution nécessite l'expression du module de Young en fonction du temps. Vu la restriction de ce modèle, viscoélasticité linéaire, aucun travail expérimental n'a été réalisé pour confirmer ou infirmer cette théorie.

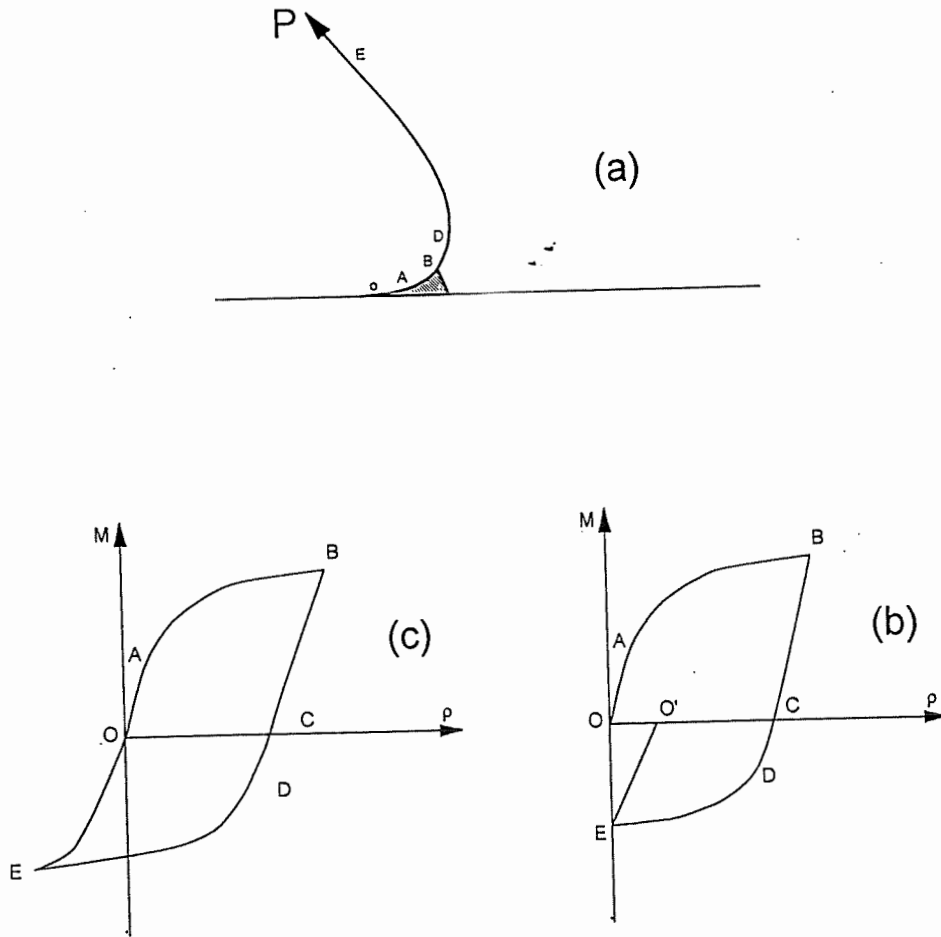


Figure 1.22: (a) Configuration de l'adhérent durant le pelage et le diagramme moment-courbure correspondant pour (b) matériau élastique-plastique, (c) matériau élastique-viscoélastique.

1.3.3. Conclusion.

Nous avons présenté une vue d'ensemble des théories de pelage avec les différents concepts dépendement du mode de déformation (élastique, viscoélastique et plastique). Cependant, le papier ne peut pas être caractérisé dans un seul de ces trois modes car:

a) le papier peut fléchir élastiquement pour les faibles courbures mais inélastiquement pour le reste,

b) plusieurs hypothèses utilisées dans ces modèles ne sont pas vraies dans le cas du papier. Par exemple, le fait de supposer que les propriétés en traction et en compression sont identiques n'est pas valable, et

c) il existe des problèmes spécifiques au papier, tels que l'effet de l'hétérogénéité et de l'humidité.

Le type de pelage (élastique ou inélastique) qu'adopte le papier a fait l'objet du premier chapitre. Nous avons pu caractériser les différentes contributions énergétiques impliquées dans le pelage d'échantillon de papier laminé. Ces derniers ont été catégorisés en énergie de détachement, W_a , (nécessaire à la rupture) et en énergie de déformation, W_d , (dissipée dans la flexion). Dans le deuxième chapitre, nous avons utilisé la décomposition de la propagation de la flexion en «flexion» et «flexion inverse» et les courbes contrainte-déformation en traction et compression pour déterminer la variation de l'énergie dissipée durant la propagation de la flexion avec la courbure. Le mode de variation ainsi trouvé a été utilisé pour la séparation de l'énergie de détachement, de

l'énergie de déformation dissipée dans la flexion. Dans le troisième chapitre, nous avons déterminé les paramètres qui peuvent modifier l'énergie de détachement et l'effet de cette dernière sur l'énergie de déformation. Dans le quatrième chapitre, nous avons étudié l'effet du raffinage sur l'énergie de détachement et l'énergie de déformation.

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CHAPITRE 2
ANGLE-DEPENDENT DELAMINATION OF PAPER.
Part I: INELASTIC CONTRIBUTION

**ANGLE-DEPENDENT DELAMINATION OF PAPER.
Part I: INELASTIC CONTRIBUTION**

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ABSTRACT

An analysis of the peel force and work for various thicknesses of laminated paper samples as a function of peel angle has been carried out. In all cases, the work of peeling, W_p , was found to depend upon the peel angle, θ , increasing as θ increases. This effect is attributed to the energy dissipated, W_d , on flexing during separation of the top and bottom layers along the line of detachment. The observations that the top layer curls after delamination, and that W_p increases with peel speed, indicate that W_d is due to both viscoelastic and plastic deformations. Both of these contributions to the overall energy dissipation increase with θ .

2.1. INTRODUCTION

The structure and properties of fibre-fibre bonds are essential factors affecting the mechanical, optical, thermal, and electrical properties of paper sheet. The fibre-fibre bond can be characterized by the bond strength (in $\text{N}\cdot\text{m}^{-2}$), which is defined as the force required to cause the failure of a single fibre-fibre bond per unit bonded area of that bond, or by the bond toughness (in $\text{N}\cdot\text{m}^{-1}$), which is the work needed, during crack propagation, to pass from the bonded to detached state per unit bonded area [1]. However, the term "bond strength" is sometimes used for both of these values in the literature [2,3].

The measured values of bond strength and of bond toughness are different when the force is applied in the plane of the bond (shear), or normal to the bond plane (tension), with the former giving higher values [1]. The shear and tension properties of the bond are also manifested during the deformation of paper sheets. When the tensile force is applied in the x-y plane (plane of the sheet), the fibre-fibre bonds are exposed to various shear stresses transferred by the fibre network. In z-direction tension, the fibre-fibre bonds are generally subjected to a tensile stress. Since most bond areas are roughly parallel to the faces of the sheet, a tensile test that pulls paper apart in the thickness direction should characterize the extent and quality of the bonding. Consequently, we will refer only to z-direction properties in what follows.

The bond strength cannot be determined from the sheet strength, which is defined as the force per unit area causing the failure of the sample. During the z-direction tensile test, the bonds are loaded successively. Therefore, the number and the area of bonds

involved in the fracture process at the failure point cannot be readily determined [4,5]. In contrast, the bond toughness may be determined from the work of detachment, which is the energy needed to separate the sample into two layers [6]. However, this method presents some difficulties. Stress concentration causes bond failure at a load lower than that corresponding to the bond failure stress [7,8]. Therefore, the correlation between the bond toughness and work of detachment is not precisely defined.

The peel test has been proposed by Skowronski and Bichard [2,9] as an alternative method for determining the bond toughness. However, these authors used the name "specific bond strength" to designate what we call "bond toughness" here. In peel tests at low speed they observed the breaking of fibre-fibre bonds but did not observe the breakage of fibre walls. They assumed that the work of peeling, W_p , is a measure of the work of detachment, W_a , which is the bond toughness times the total area created during bond failure. Retulainen and Ebeling [3] found that the peel test gave results which were not in agreement with the generally accepted effect of beating. Consequently, they suggested that stress geometry and bending during delamination affected their results. Stratton [1] proposed that, for a 90° peel angle and relatively thin samples ($60 \text{ g}\cdot\text{m}^{-2}$), the in-plane shear stress component and the work of bending in the region of delamination should be negligible. Consequently, W_p and W_a should be equal. As a result, he reported that the peel test is more sensitive to the modification of interfiber bonds by beating, wet pressing and presence of fines than the ultrasonic test in the z-direction. The peel test appears to be a convenient method for the determination of bond toughness in paper. However, there is no detailed treatment in the literature of the effect of different

mechanisms involved in paper delamination upon the work of peeling .

It is known from research on adhesives and plastics that peeling is capable of giving reproducible and reliable results, which provide suitable information on adhesion, interface bonding, sticking, etc. However, the interpretation of these results is difficult for some materials. The relationship between the measured peel force (or work of peeling) and work of detachment depends upon the mechanical and rheological properties of the sample. In addition, the stress geometry and bending of the detached layer can affect the delamination process.

The peel process can be classified as elastic or inelastic depending upon the nature of the adherent. In an ideal case of elastic peeling, when the detached layer deforms only elastically, the peel force is inversely proportional to $1-\cos\theta$, where θ is the peel angle, and is a direct measure of detachment work [10-13]. The first theoretical analyses of the elastic peeling process were presented by Spies [10] and by Bikerman [11]. Following this, several analyses of experiments, based on the elastic peeling model, appeared in the literature [14-18]. However, it was found that in many experiments the peel force does not follow a $(1-\cos\theta)^{-1}$ law. This discrepancy was attributed to the extensibility of the adherend [19], the effect of thickness of the adhesive layer [16,18], stress distribution in the crack zone and the change from shear stress to cleavage stress in the crack zone [14,20,21].

Gent and coworkers [22,23] attributed the observed disagreement to inelastic peeling. The presence of permanent residual curvature after delamination indicates the contribution of plastic bending deformation to the delamination process [23]. This

phenomenon demonstrates that the detached layer deforms inelastically, in that some energy is spent on this deformation. It was shown that the peel force increases with bending curvature and presents a maximum as a function of the thickness of the detached layer. This was attributed by the authors to the dissipation of energy in plastic bending.

For materials showing inelastic peeling behaviour, angle- and rate-dependent delamination is difficult to analyze. However, it provides valuable information about the plastic or viscoelastic properties of the detached layer. The viscoelastic properties of samples have been revealed by the effect of peel speed on the peel force as well as by the presence of relaxation phenomena [24-26]. Recently, Loukis and Aravas [27,28] have presented a general formulation of the problem of peeling a linear viscoelastic film from a rigid substrate. In summary, both work of detachment and work of deformation contribute to the total work of peeling. Their contributions depend upon the mechanical properties of the samples studied.

This study concerns the relation between W_a and W_p in the case of paper. We demonstrate here that the deformation work in the bent delaminated layer of paper has an important effect on the measured value of peel force. The effects of detached layer thickness, peel angle and peel rate on the peel force are reported.

2.2. EXPERIMENTAL

A schematic presentation of the peel test is shown in Figure 2.1. In this configuration, the top layer (A) undergoes flexion during peeling at a given angle, θ . The

bottom layer (B), glued to a rigid substrate (C), maintains a flat shape during the process.

In one series of experiments, paper was delaminated at peel angles between 90° and 170° at a constant peel rate, 2.54 cm min^{-1} , using an IMASS Model SP-103B Slip/Peel tester. In a second series, the peel rate was varied from 0.5 to 25 cm min^{-1} . The peel force was recorded and its mean value determined (calculated as the area under the curve divided by the length of peeled material). All experiments were performed at room temperature and at 50% relative humidity.

The measurements were performed on handsheets prepared from bleached softwood kraft pulp. The samples were made by lamination of two handsheets of varying basis weight (from 30 to 60 g m^{-2}) from pulp beaten to 500 revolution in a PFI mill. Lamination was produced using wet pressing at standard pressure (420 kPa). These laminated handsheets were used so that the thickness of the detached layer could be controlled, and to prevent entanglement between fibres at the interface. In this way, fibre breaking and multilayer delamination were avoided.

Test samples of width (b) and length (l), $2 \text{ cm} \times 15 \text{ cm}$ ($b \times l$), were attached to a metal support using double sided pressure sensitive adhesive tape (3M). The top layer was reinforced using one-sided adhesive tape (3M). In order to achieve uniform and reproducible contact between tape, paper and metal plate, test samples were pressed between metal plates for 5 min under a pressure of 1.75 MPa at room temperature. Following that, the samples were conditioned at 50% relative humidity for at least 24 hours before the test.

2.3. RESULTS

2.3.1. Effect of peel angle

Fig. 2.2a shows typical variation of peel force per unit width (P/b) with θ for a laminated handsheet (30/30 $\text{g}\cdot\text{m}^{-2}$) (30/30 means 30 $\text{g}\cdot\text{m}^{-2}$ for the top layer(A) and 30 $\text{g}\cdot\text{m}^{-2}$ for the bottom layer(B)). The observed U-shaped curve shows a broad minimum ($120^\circ \leq \theta \leq 140^\circ$). Similar curves were obtained in the peel test for all paper investigated. It appears more relevant to present the results in the form of peel work W_p as a function of the peel angle.

$$W_p = \frac{P}{b} (1 - \cos\theta) \quad (1)$$

We observe that W_p increases as θ increases (Fig. 2.2b). Similar results for various materials [12,14,29] have been reported in the literature.

2.3.2. Effect of thickness

It has been reported that the thickness of the layer which undergoes irreversible flexion affects the peel force [23,29]. The effect of the top layer thickness on the peel force was explored using laminated handsheets that varied in basis weight from 20/60 to 60/60 g m^{-2} . The variation of the thickness with the basis weight and the effect of the top

layer thickness on the peel force at 90° are shown in Table 2.1.

Table 2.1: Variation of thickness with basis weight, and effect of the top layer thickness on the peel force.

Basis weight A/B (g m⁻²)	Total thickness (μm)	Top layer thickness (μm)	Peel force at 90° (N m⁻¹)
20/60	140,5	46,3	33,94
25/60	148,3	54,2	35,73
30/60	150,9	62,0	38,92
40/60	171,8	77,7	44,36
50/60	193,5	93,4	49,95
60/60	203,3	101,7	57,67

Fig. 2.3 shows the peel work, W_p , versus the thickness (t) of the top layer at three different angles. The experiments show an increase of W_p with t for all angles. This increase is more pronounced for large angles than for small. These observations suggest a relationship among thickness, peel angle and peel work.

2.3.3. Plastic deformation

When the delamination was stopped, the detached part was cut near the detachment zone and released from the clamp of the peel tester. It curled into a tight spiral, as shown in Fig 2.4. This process took place in two steps. The first was instantaneous curling, the curvature passing from zero to a given value. The second step was slow curling, with the curvature continuing to increase with time. This can be described as strain relaxation. The process was always complete after one hour. The final curvature of paper near to the detachment zone was higher than that over the rest of the sample. In what follows, this highest curvature is called the residual curvature ρ_{res} . Fig. 2.5 shows that ρ_{res} increases with θ . This behaviour was observed for all samples studied; i.e. for all beating levels, wet pressing conditions and thicknesses.

2.3.4. Effect of peel speed

The effect of speed was observed during and after the peeling process. The effect of speed upon work of peeling, W_p , is shown in Fig. 2.6. The increase of W_p with speed depends upon θ , it being clear that the effect of speed is highest at large angles.

After peeling, two effects of speed were observed: the first, just at the end of the delamination process and the second, after the detached layer was released from the clamp. Force relaxation began immediately after peeling was interrupted. This relaxation

was associated with a change in the bending curvature. Thus, the relaxation process originates, at least partially, in the bent part. In order to measure the relaxation process, we used the difference between the initial ($t=0$ min) and final ($t=6$ min) values of the force, normalized to the initial value, $\Delta P/P$. Fig. 2.7 shows that $\Delta P/P$ increases with both angle and peel speed.

The residual curvature ρ_{res} observed when the detached layer was released from the clamp decreased with increasing peel rate, as shown in table 2.2. A similar phenomenon was observed by Sancaktar [26] and was attributed to the viscoelastic nature of the detached strip. The relaxation of the bent region changes with the peel speed as do ρ_{res} and W_p , especially at large angles.

Table 2.2. Effect of peel rate upon work of peeling W_p and residual curvature ρ_{res} for samples prepared from laminated handsheet 30/30 g m². The peel angle was 90°.

Peel rate, cm/min	0,5	2,5	5,1	12,7	25,4
W_p, N·m⁻¹	69,6	71,0	75,6	87,2	91,8
ρ_{res}, m⁻¹	84,0	71,4	69,0	62,5	64,5

2.4. DISCUSSION and CONCLUSIONS

The top layer is under flexion during peeling. This flexion leads to the curling of the detached layer. Knowing that passage from the flat to the curled state requires an expenditure of energy [30], we may conclude that energy is dissipated in flexion during peeling. Thus, we conclude that paper undergoes inelastic peeling behavior. The work of peeling, W_p , is the sum of the work of detachment, W_a , dissipated in the separation of the bottom from the top layer, plus the work of deformation, W_d , dissipated irreversibly in flexion.

$$W_p = W_a + W_d \quad (2)$$

The energy spent to curl the top layer is a plastic contribution. The increase of the residual curvature of the curled layer with θ shows that this plastic deformation energy also increases with θ .

Another contribution to the deformation energy, viscoelastic deformation, is indicated by the effect of the speed, and by the presence of relaxation. In fact, the variation of work of peeling with speed in other materials has already been explained as due to viscoelastic phenomena [25,26]. The relaxation phenomenon observed in the bent region and the decrease of ρ_{res} with speed indicate that the viscoelastic dissipation is localised in the region under flexion. Furthermore, the effect of the peel speed upon work

of peeling, which increases with increasing θ , reflects the increase of viscoelastic dissipation with θ .

Thus, we may satisfactorily account for the general nature of the bending dissipation in terms of viscoelastic and plastic contributions and their variation with the peel parameters. The increase of W_p with the thickness of the top layer (the layer which undergoes flexion) and its increase with θ can be attributed to the work of deformation. This is in agreement with previously published work [23,29]. This deformation energy is always present during peeling, even for small thicknesses of removed layer. This shows that the bending work is an important contribution to the peel work for paper, and may not be neglected, as was done earlier [1,2,31]. A general formalism for analysis of the peel process and the results of such analyses are presented elsewhere [32].

2.5. ACKNOWLEDGMENTS

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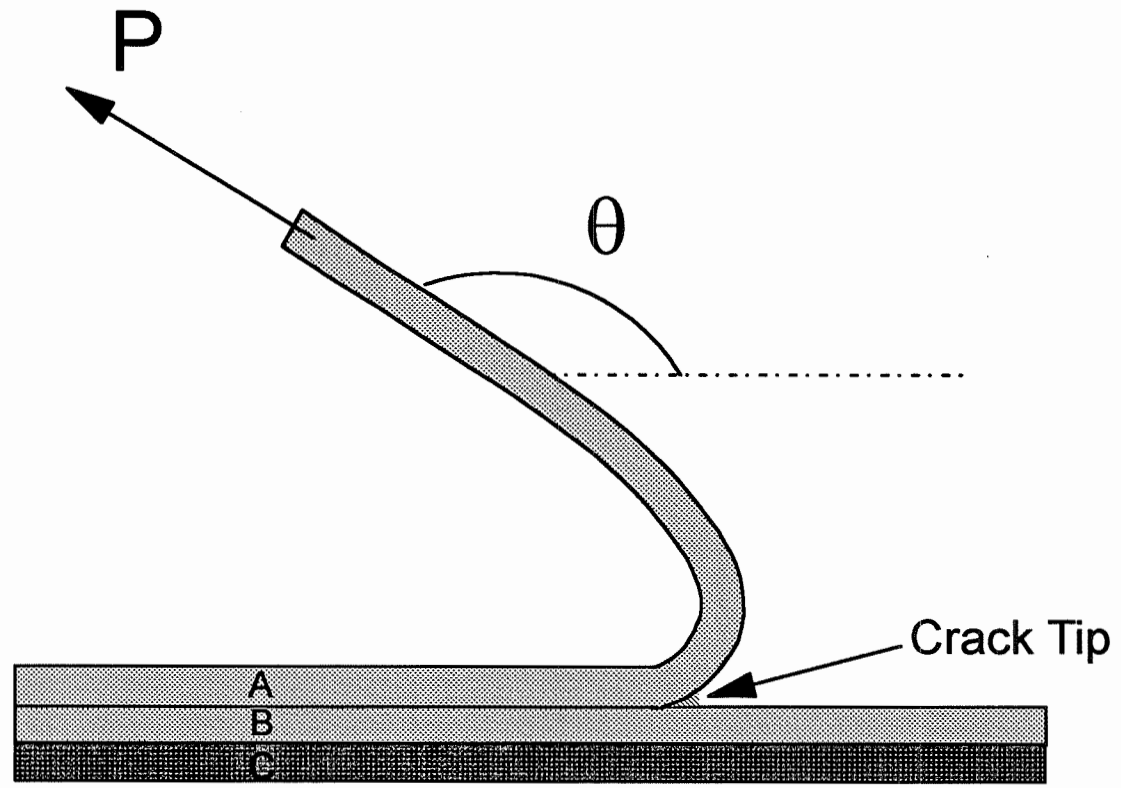


Figure 2.1. Schematic representation of the configuration for the peel experiments

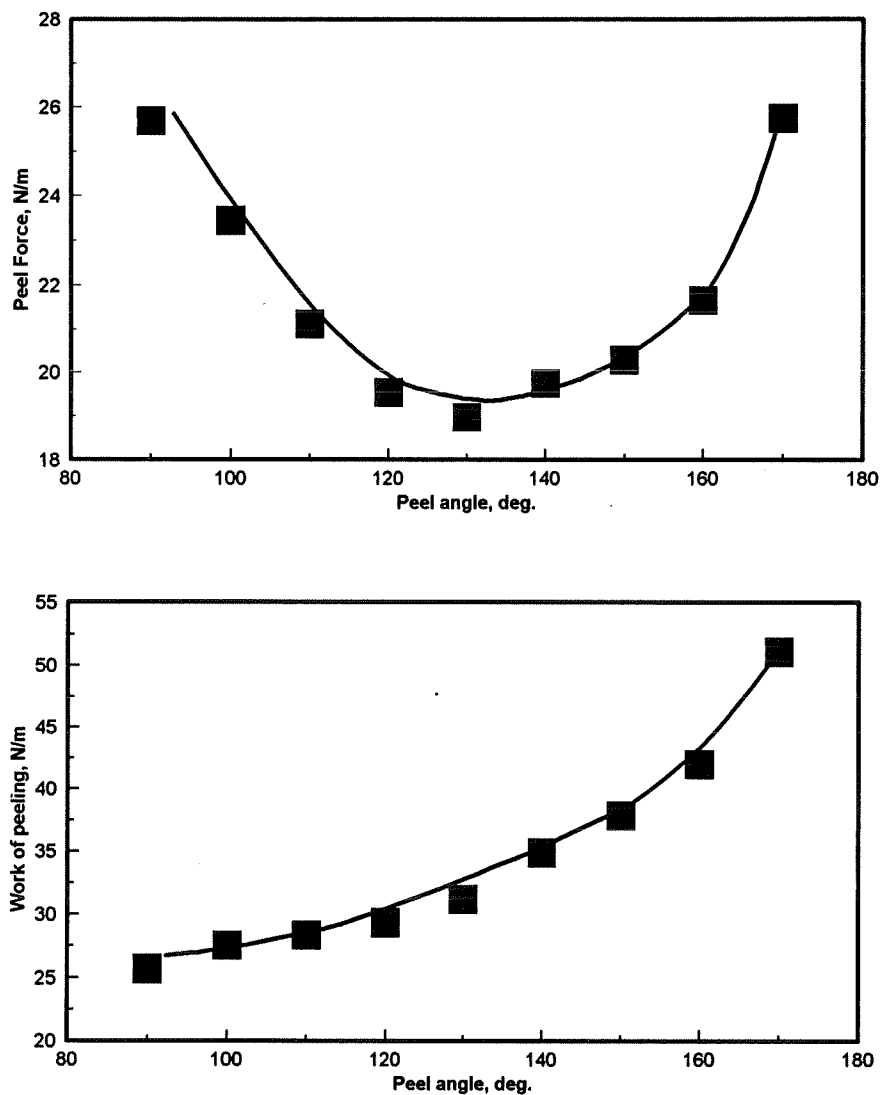


Figure 2.2. The peel force per unit width, P/b , (a) and the calculated work of peeling, W_p , (b) as a function of peel angle, θ , for laminated samples prepared under standard conditions ($30/30 \text{ g/m}^2$, wet pressing at a pressure of: 420 kPa; beating level: 500 PFI; peel rate: 2.54 cm/min).

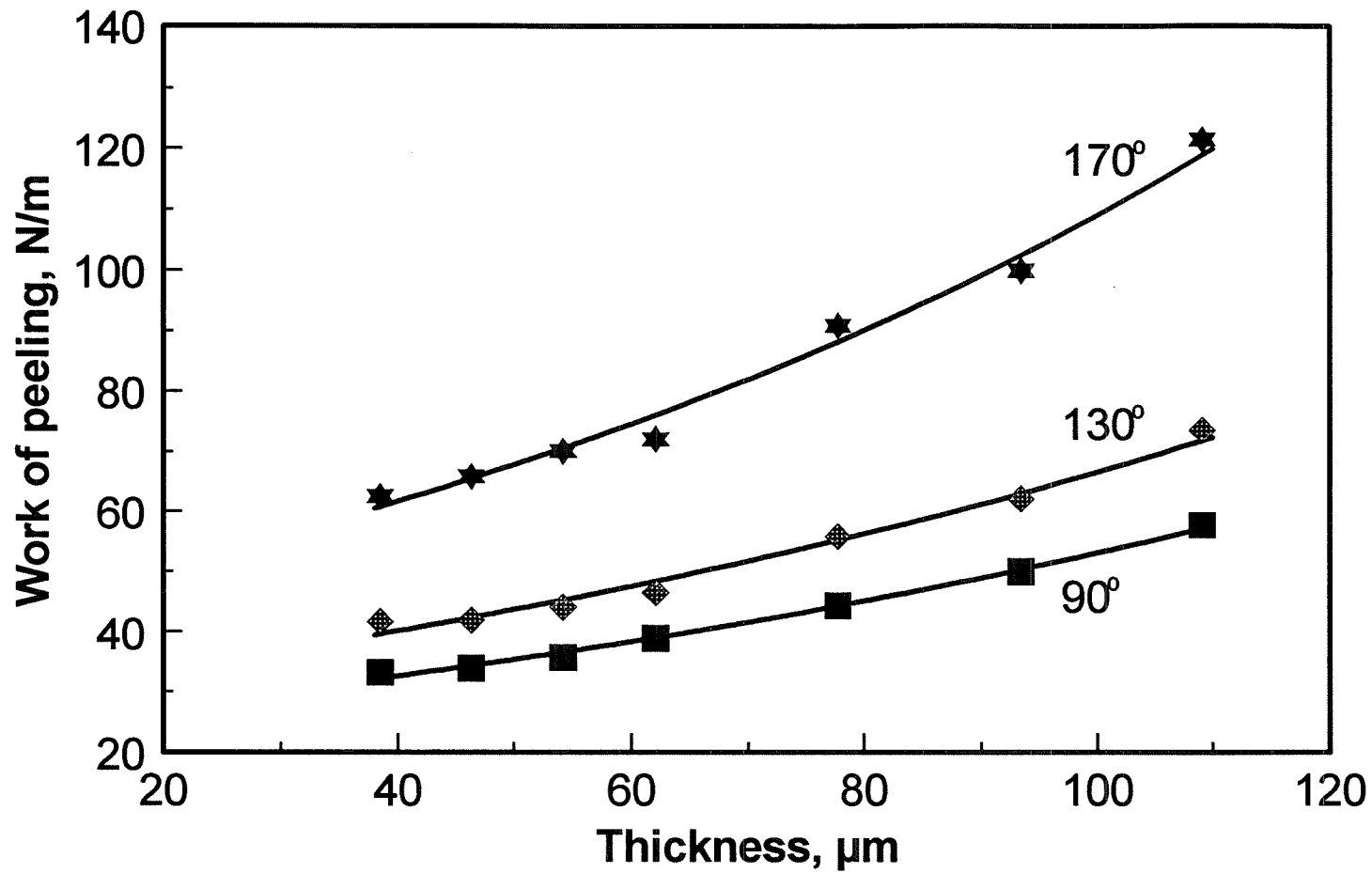


Figure 2.3. The dependence of work of peeling (W_p) on the thickness of the upper layer delaminated from a substrate of constant thickness (basis weight between 20/60 and 60/60 g/m^2). The samples were prepared by wet pressing at a pressure of: 420 kPa; beating level: 500 PFI; peel rate: 2.54 cm/min.

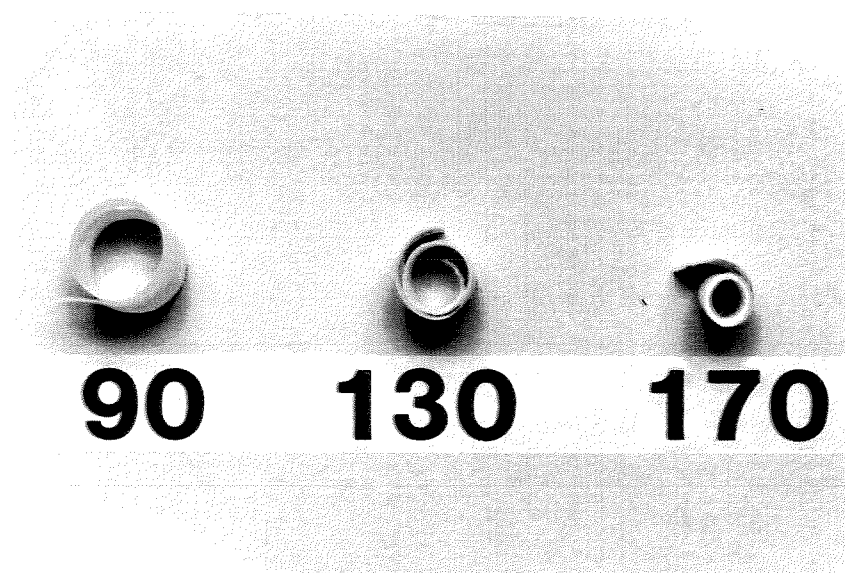


Figure 2.4. Photo of the upper layer after delamination. The numbers indicate the peeling angles.

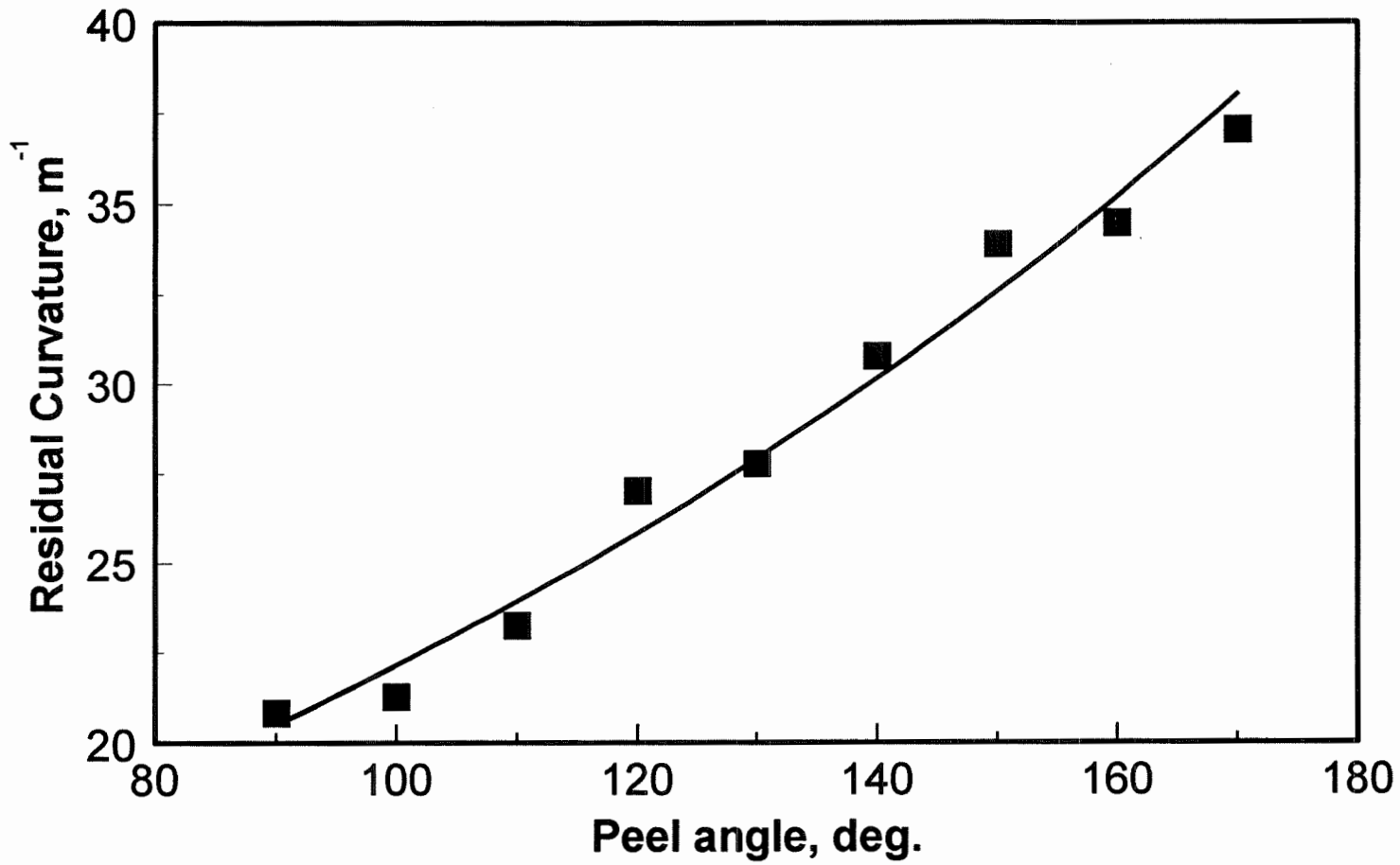


Figure 2.5. The variation of the residual curvature of the detached layer as a function of peel angle.

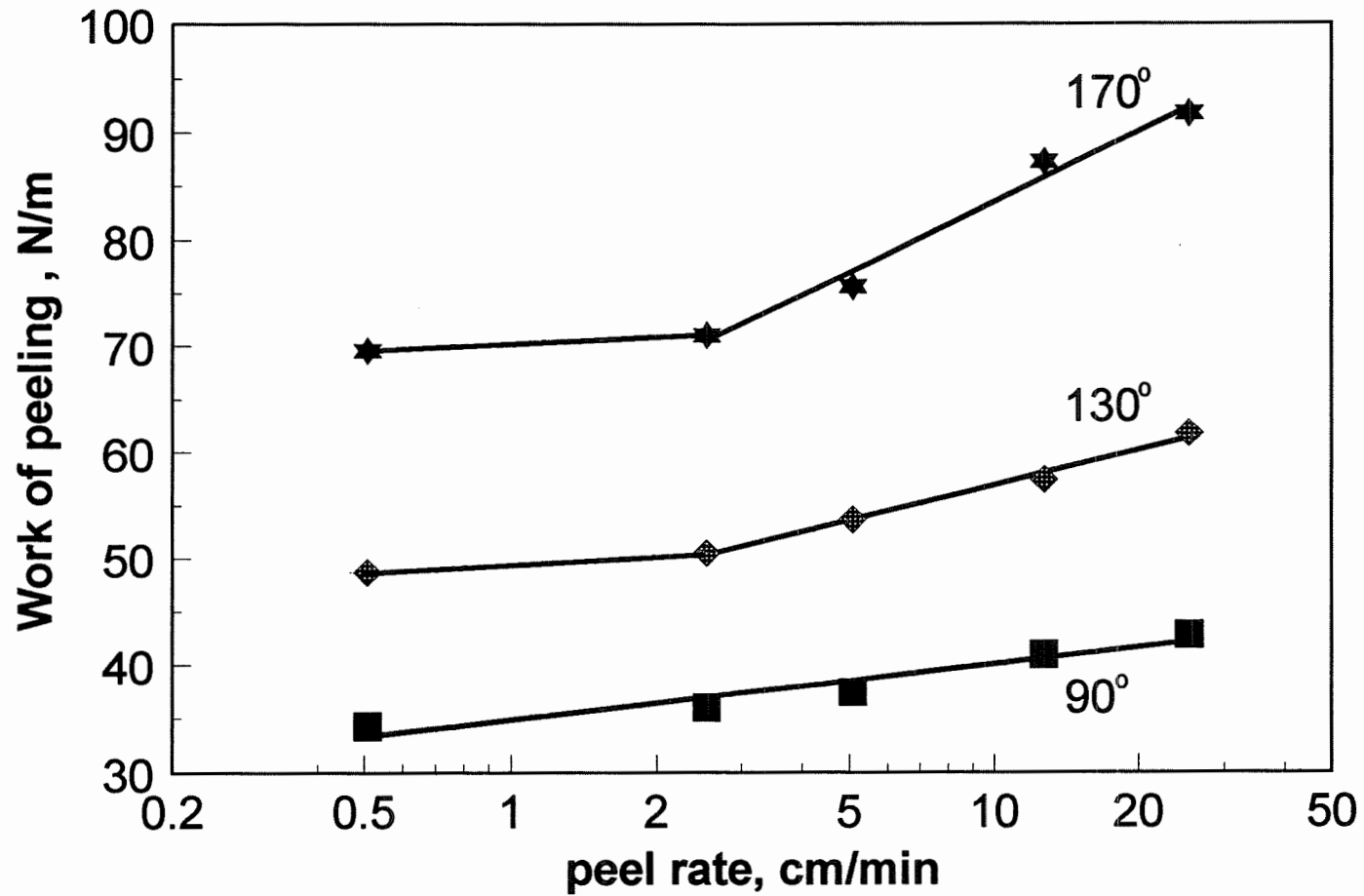


Figure 2.6. The dependence of work of peeling (W_p) upon peel rate for laminated samples ($30/30 \text{ g m}^{-2}$, wet pressing at a pressure of: 420 kPa; beating level: 500 PFI) for various angles, θ .

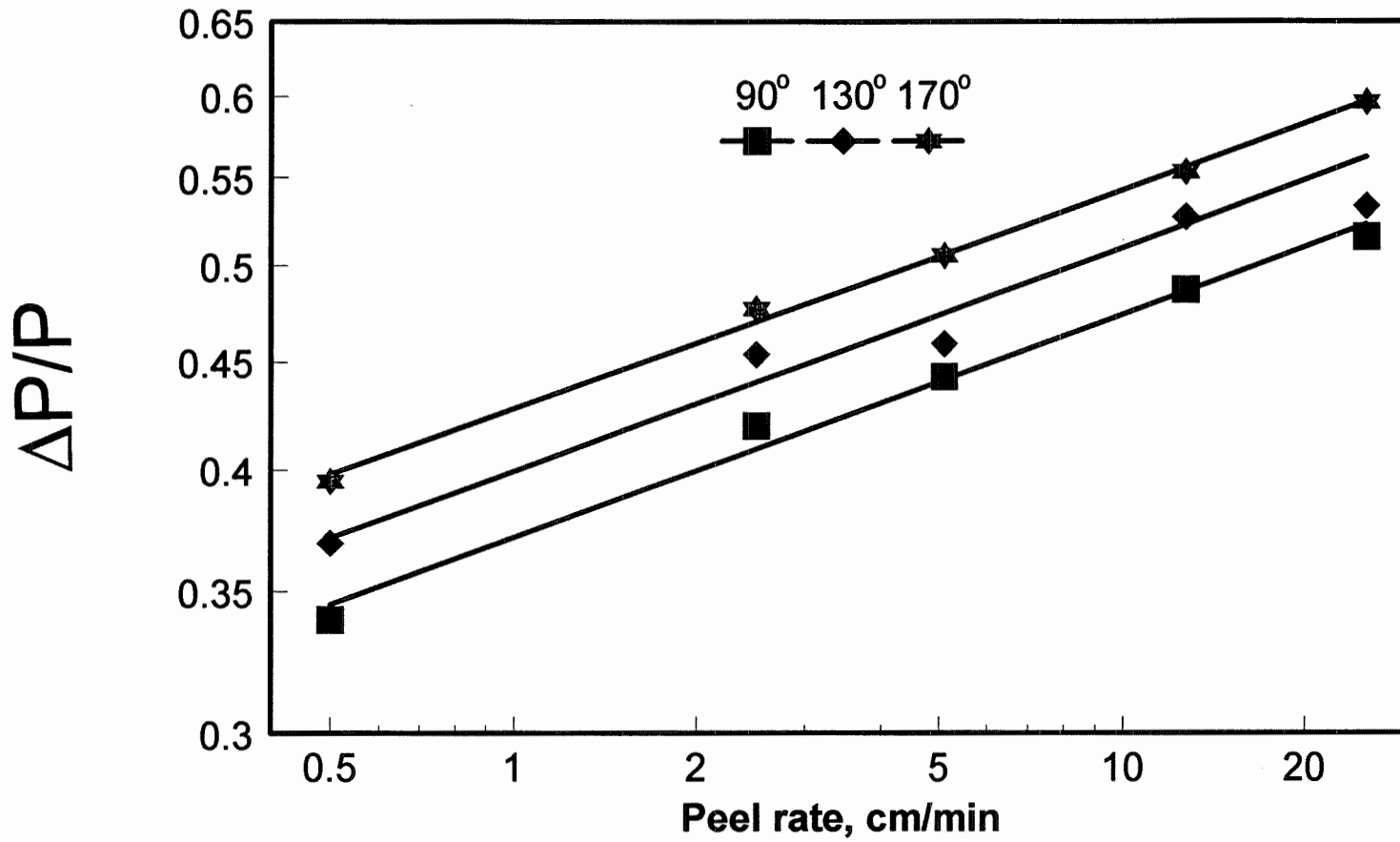


Figure 2.7. Normalized relaxation force vs peel rate for laminated samples (30/30 g/m², wet pressing at a pressure of: 420 kPa; beating level: 500 PFI) for various angles, θ .

CHAPITRE 3**ANGLE-DEPENDENT DELAMINATION OF PAPER.
PART II: DETERMINATION OF DEFORMATION AND
DETACHMENT WORK IN PAPER PEELING**

**ANGLE-DEPENDENT DELAMINATION OF PAPER.
PART II: DETERMINATION OF DEFORMATION AND
DETACHMENT WORK IN PAPER PEELING**

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Abstract

Laminated handsheets of paper were peeled apart at various angles. The work of peeling increased with the peel angle. An analysis has shown that the work of peeling consists of two main contributions: work of detachment and work of inelastic deformation. The work of deformation expended in the bending and unbending modes was quantified using a theoretical model based on compressional and tensional stress-strain characteristics. Application of this model leads to the conclusion that the work of detachment is independent of angle.

3.1. INTRODUCTION

The peel test, schematically presented in Fig.3.1, is widely used to investigate the properties of adhesives and of interfaces. In the figure, part B is rigid or is attached to a rigid support; part A is peeled away. In the case where they are different or are held together by an adhesive, A and B are called adherend and substrate, respectively. When a sample, such as paper, is separated into two pieces, A and B are denoted as the top and bottom layers. In what follows, the energy required for separation at the crack tip is called the "work of detachment", W_a , the total work spent during the peel test is the "work of peeling", W_p , and the energy spent in bending and unbending part A is the "work of deformation" W_d .

The variations of work of peeling with peel angle, peel rate and the thickness of the top layer [1,2] introduces substantial difficulties in the analysis of the results and particularly for the determination of the toughness at the crack tip. It is widely recognized that in a peeling experiment the detached layer may undergo irreversible deformation in the bent region. Therefore, energy balance approaches to peeling have concentrated on determining the energy consumed by plastic and viscous processes due to the propagation of the bend in the detached layer during separation. This is the work of deformation, W_d . The history of this problem, and of the attempts to resolve it, are presented in detail elsewhere [2].

Gent and co-workers [3,4] were the first to develop an experimental method to determine W_d and its variation with film thickness and curvature in flexion. Their results

were in agreement with the predicted values of W_d , calculated using elementary beam bending theory in an ideal elastic-plastic strip. Aravas and coworkers [5,6] investigated the flexion propagation as a cycle of bending and unbending, and considered ideal, elastic-plastic, strain hardening and linear viscoelastic materials. Mantel and Descaves [7] found that the deformation energy for an elastic-plastic material depends upon the work of detachment and the speed. For the case of bimaterial strips in which at least one material exhibits viscoelastic behaviour, Sancaktar [8] has developed a theoretical method for determining the radius of curvature and the flexural moment as functions of the thicknesses of adhesive and its backing, and of Young's moduli. Recently, Farris and Goldfarb [9] used deformation calorimetry to measure the energy stored as latent internal heat in poly(ethylene terephthalate) during peeling. They showed that the fraction of the energy which is dissipated as heat increases with peel rate and varies with the mechanical properties and thickness of the backing.

Despite extensive investigations of bending propagation during peeling, the determination of the work of deformation consumed during bending propagation is still not complete. In particular, all mechanical analyses of bending have assumed that the stress-strain behaviour is identical in tension and in compression. Consequently, the neutral surface is fixed at the midline of the adherend layer [3,7]. However, there is a difference between compressional and tensional stress properties of materials. As a result, the neutral surface must shift with the curvature, thus affecting the bending moment and work. Thus, the relation between the work of detachment, bending curvature and the peel angle has not been thoroughly analyzed in the literature.

The determination of W_d for materials like paper requires a complete stress analysis, since paper exhibits both plastic and viscoelastic dissipation [2]. In addition, stress-strain curves and flexural moments are affected by sheet anisotropy [10]. In what follows, we shall use stress-strain data in tension and compression to determine bending propagation properties, such as the flexural moment and deformation work, and their variations with curvature. The results will be used to determine the work of detachment, W_a , and to describe the effect of the peel angle and the thickness of peeled strips on work of peeling, W_p .

3.2. EXPERIMENTAL PROCEDURE

3.2.1. Sample preparation

The handsheets to be investigated were prepared in two ways, depending upon the aim of the experiment. Bleached kraft pulp (Q90) beaten for 500 revolutions in the PFI mill was used for all experiments. For the peel test, handsheets were prepared by lamination, by standard wet pressing of two individual sheets which had never been dried. The basis weights of the resulting handsheets were 30/30 g m⁻² (30/30 g m⁻² means 30 g m⁻² for the top layer (A) and 30 g m⁻² for the bottom layer (B)). This method assures a constant thickness of the detached layer, prevents fiber entanglement at the interface, and minimizes bulk delamination. The same samples as those prepared for delamination were used for tensile stress-strain analysis. Compression stress-strain studies were carried out on sheets prepared under standard conditions, but having 300 or 400 g/m² basis

weight [11].

3.2.2. Apparatus and measurement procedure

The delamination test was performed on the IMASS, model SP-103B Slip/Peel Tester equipped with a high resolution video recording system. This system permitted the detailed microscopic observation of the delamination process. The basic experimental method is illustrated schematically in Fig.3.2. A paper sample, 2cm wide and 15cm long, was carefully glued to a metal plate mounted on the peel tester, using double sided Scotch tape. Experiments were performed for peel angles between 90° and 170° . The peel rate was kept constant at 2.54 cm/min. During the test, a computerized video camera was used to record and store images on a hard disk. The maximum curvature was determined from the radius of the circle fit to the video image of the delamination zone.

Stress-strain measurements in tension and in compression were performed on an Instron model 1011 tensile tester. The system was equipped with a computer interface, which is used to control the Instron and for data acquisition and processing. The samples dimensions of were 1.5cm by 10cm for tensile measurement and 1.5cm by 7mm for compression tests (using heavy clamps, made for this experiment) . The test speed was fixed at 2 mm/min .

3.3. ANALYSIS OF THE PEEL MECHANISM

The energy balance, for steady state peeling of a flexible but inextensible strip, can be written as [2]:

$$W_p = \frac{P}{b} (1 - \cos\theta) = W_a + W_d \quad (1)$$

In Eq. 1, P/b is the peel force per unit width, and θ is the applied peel angle.

3.3.1. Energy dissipated during bending and unbending of the paper

The elementary work per volume element performed during deformation is given by the expression [7]:

$$\delta\Phi = [\sigma_{ij}] [\delta\epsilon_{ij}] , \quad (2)$$

where $[\sigma_{ij}]$ is the stress matrix and $[\delta\epsilon_{ij}]$ is the differential strain matrix.

In the case of a sheet subject to bending or unbending as shown in Fig. 3.3, and assuming that $\sigma_{xz} = \sigma_{zz} = 0$,

$$\delta\Phi = \sigma_{xx} \delta\epsilon_{xx} . \quad (3)$$

The strain is given by:

$$\epsilon_{xx} = \frac{L - L_0}{L_0}, \quad (4)$$

where L_0 is the initial length for a given segment, L is the length at a given stress due to the bending. To express ϵ in term of radius and z -coordinate, we must introduce the neutral surface as the origin of the z coordinate.

The problem is that the neutral surface shifts due to the difference in stress between tension and compression. When the neutral surface moves to a region which was previously stretched, the length of the segment initially L_0 , becomes L_1 through plastic deformation. Hence, the introduction of L_1 in the expression for the strain is helpful in the determination of ϵ as a function of z and of the radius:

$$\epsilon_{xx} = \frac{L - L_1}{L_1} \cdot \frac{L_1}{L_0} + \frac{L_1 - L_0}{L_0} = \frac{z}{R} \cdot (\epsilon_p + 1) + \epsilon_p, \quad (5)$$

where $\epsilon_p = (L_1 - L_0)/L_0$ is the plastic deformation at the neutral surface. Skowronski and Robertson [12] demonstrated that $\epsilon_p/\epsilon = 0.06$ for a beaten kraft paper at a tensile stress-strain values $\sigma = 60\text{MPa}$ and $\epsilon = 1.11\%$. This condition may be seen as an upper limit for a neutral surface, since there is relatively small stress development in the region of the neutral surface during bending and unbending. Thus ϵ_p may be neglected without significant error. Consequently, in the case of bending ϵ_{xx} and $\delta\epsilon_{xx}$ are given by:

$$\epsilon_{xx} = \frac{z}{R} = z\rho, \quad (6)$$

and

$$\delta \epsilon_{xx} = z \delta \left(\frac{1}{R} \right). \quad (7)$$

In Eqs. (6) and (7), R and ρ are the radius and curvature, respectively, and z is the coordinate measured in the thickness direction from the neutral surface, as shown in Fig. 3.3.

In the case of unbending, ϵ_{xx} and $\delta \epsilon_{xx}$ are given by:

$$\epsilon_{xx} = \frac{z \left(\frac{1}{R} - \frac{1}{R_0} \right)}{1 + \frac{z}{R_0}} \approx z \left(\frac{1}{R} - \frac{1}{R_0} \right) = z (\rho - \rho_0), \quad (8)$$

and

$$\delta \epsilon_{xx} = z \delta \left(\frac{1}{R} - \frac{1}{R_0} \right) = z \delta (\rho - \rho_0), \quad (9)$$

where R_0 and ρ_0 are the initial radius and curvature respectively.

The work per unit area is represented in bending by:

$$W_b = \int_{\rho} \left[\int_{-h}^h \sigma_{xx}(z) z dz \right] d(\rho) = \int_{\rho} M_b(\rho) d(\rho), \quad (10)$$

and in unbending by:

$$W_{ub} = \int_{\rho} \left[\int_{-h}^h \sigma_{xx}(z) z dz \right] d(\rho - \rho_0) = \int_{\rho} M_{ub}(\rho - \rho_0) d(\rho - \rho_0), \quad (11)$$

where

$$M_b(\rho) = \int_h \sigma_{xx}(z) z dz, \quad (12)$$

and

$$M_{ub}(\rho - \rho_0) = \int_h \sigma_{xx}(z) z dz. \quad (13)$$

M_b in Eqs. (10) and (12), is the bending moment per unit width, M_{ub} in Eqs. (11) and (13), is the unbending moment per unit width, and h is the thickness.

As shown in Fig.3.3, the origin for the calculations of moment and work, in Eqs. (10) - (13), is the intersection between the z (radial) axis and the neutral surface. Determining the origin requires determining the position of the neutral surface, which is defined as the plane where the sum of the stress in the x -direction is zero. Consequently, the differential dimensional change of this surface is approximated as zero (neglecting the plastic deformation). Equilibrium of stress is used in order to determine the position of the neutral surface in the thickness for any given curvature:

$$\int_h \sigma(z) dz = 0. \quad (14)$$

3.3.2. Bending mechanism

The analyses of the curvature and deformation have been discussed by Igarashi [13] and by Aravas et al. [5]. A schematic representation of the peel test geometry (a) and

the corresponding moment vs curvature diagram (b) are shown in Fig. 3.4. The segments OA and AB correspond to elastic (beginning of bending) and inelastic deformation of the strip, respectively. The maximum curvature ρ_{\max} occurs at point B. With the propagation of peeling, the sheet unbends, first elastically in the segments BD and then inelastically in DE. Zero curvature is reached at the point E. However there is a residual stress at this point. When the grips of the peel tester are released, the load is removed, producing the permanent curvature, ρ_{res} [2] (point O'). The area of the hysteresis loop (OABCDEO') represents the deformation work, W_d , the energy dissipated in bending and unbending during the peel test.

In that follows, we divide the analysis of the hysteresis loop, OABCDEO', by considering two loops, OABC and CDEO'. The loop OABC results from the bending process, its area representing the bending work, W_b . The loop CDEO' results from the unbending process, its area representing the unbending work, W_{ub} . In this case, we plot the absolute value of the unbending moment, $|M_{ub}|$, and W_{ub} versus ρ' ($\rho' = \rho_0 - \rho$) instead of the real curvature (ρ), as in Eqs. (11) and (13). The shape of $|M_{ub}|$ vs ρ' will be similar to that observed for bending.

3.4. RESULTS AND DISCUSSION

Consider a cross-section of paper sheet subjected to bending, as shown in Fig.3.3. As $z=0$ corresponds to the neutral surface, the material at $z>0$ is under tension and that at $z<0$ is under compression. The bending moment, the bending work and the position of

the neutral surface depend upon the variation of stress and strain over the cross-section for each curvature. Their determinations require stress-strain data in tension and in compression.

During the unbending process, the beam is subjected to a moment of force which reduces its curvature. The part which was compressed during bending undergoes tension during unbending. The tensile stress-strain behaviour of the previously compressed region will differ from that of the virgin sample. Hence the analysis of the unbending characteristic requires knowledge of the tensile stress-strain properties of previously compressed paper.

3.4.1 Stress-strain analysis

Fig.3.5 shows the stress-strain curves for tension (a) and compression (b). For the virgin sample, the initial elastic modulus in tension (a) and in compression (b) are equal. However, the ultimate stress (strength) and strain at failure are much lower in compression than in tension. This agrees with previously published data [14,15]. After the ultimate compressive strength is reached, the stress drops to a moderate value and then fluctuates about a constant value for further strain. This process is accompanied by a pronounced deformation in the sample shape.

Curve (c) presents the tensile stress-strain results obtained for a sample which was previously compressed beyond the compression strength ($\sim 0.5\%$ strain). The mechanical

properties (elastic modulus, strength) are much lower for curve (c) than those for curves (a) and (b). It is useful to note here that the second compression of a sample which had previously been compressed and then placed under tension, exhibits a stress-strain curve similar to curve (c). Compression of a sample which had previously been under a slight tension yielded a stress-strain curve similar to curve (b).

3.4.2. Neutral surface

The position of the neutral surface is dictated by the equilibrium of stresses as presented in Eq. (14) which can be rewritten:

$$\int_0^{h_c} \sigma_c(z) dz = \int_0^{h_t} \sigma_t(z) dz. \quad (15)$$

In Eq. (15), σ_n and h_n are the absolute values of the stress (σ) and thickness (h) in compression ($n=c$) and tension ($n=t$). The sum of h_c and h_t must be equal to the thickness, h , of the detached strip (Fig. 3.3):

$$h_c + h_t = h. \quad (16)$$

Finding the position of the neutral surface is equivalent to the determination of the thicknesses h_c and h_t . In order to achieve this, the stress-strain data are processed using a computer program based on a numerical solution of Eq. (15) derived in the appendix (see Eq. (C)) where the z variable is replaced by an ϵ variable using Eq. (6) in the case

of bending. The procedure consists of using each point (σ_t, ϵ_t) from the tension stress-strain curve as the upper limit, namely $\epsilon_t = h_t/R$. By iteration, we determine the corresponding limit point in the compression stress-strain curve (σ_c, ϵ_c) at which Eq. (15) is fulfilled ($\epsilon_c = h_c/R$). That is, if (σ_t, ϵ_t) represents the tensile stress and strain at point T (Fig 3.3), then (σ_c, ϵ_c) will represent the compressional values at point C. From the values ϵ_t and ϵ_c , we can determine the ρ , h_t and h_c using Eq. (D) (see Appendix).

For unbending, the calculations are performed using the curvature variable $\rho' = \rho_0 - \rho$. Otherwise, the same procedure is used as for bending.

Fig.3.6 shows the variation of h_t and h_c vs ρ for bending and ρ' for unbending. The force balance in bending imposes the condition that h_t decreases while h_c increases with the curvature. This means that the neutral surface shifts to the convex side (the side under tension) during bending. In unbending, compression (h_c) and tension (h_t), change sides. The increase of h_c (decrease of h_t) indicates that the neutral surface moves back toward the middle of the thickness during the unbending process.

It is useful to note that a region in the middle of the top layer successively undergoes tension and compression. However, the stress-strain level reached during tension has no effect upon the stress-strain results in compression, as discussed above.

3.4.3. Bending and unbending moments

The moment (per unit width) given by Eqs. (12) and (13) can be rewritten, taking

into consideration both compression and tension:

$$M_{b,ub} = \int_0^{h_c} \sigma_c(z) z dz + \int_0^{h_t} \sigma_t(z) z dz. \quad (17)$$

Obviously the moment depends upon the position of the neutral surface. Carlsson et al [10] found that calculations for bending based on Eq. (17), are in agreement with the experimental results for paperboard. Peterson [16] has used the experimental data of the bending moment and the stress-strain curves, in compression and tension, in Eq. (17) to determine what he refers to as "the effective structural thickness". He found no significant difference between the calculated and the measured thicknesses. A numerical solution for the moment is presented in the appendix (Eq. E).

Fig.3.7 shows the calculated moment versus the curvature in the case of bending, M_b vs ρ (curve a) and unbending, M_{ub} vs ρ' (curve b). A comparison of the two curves shows that the bending moment is always higher than the unbending moment. This means that paper offers more resistance to bending than to unbending. The stiffness, S (represented by the initial slope), is also higher in bending than in unbending, $S_b > S_{ub}$.

3.4.4. Bending and unbending work

The irreversible work done in the bending and unbending of the sheet is the

difference between the total work and the elastic energy stored in bending the beam by assuming elastic-plastic behavior. It is given by:

$$\begin{aligned} W_b &= \int_0^{\rho_{\max}} M(\rho) d\rho - \left(\frac{M_b^2(\rho_{\max})}{2S} \right) \\ W_{ub} &= \int_0^{\rho_0} M(\rho') d\rho' - \left(\frac{M_{ub}^2(\rho_0)}{2S_{ub}} \right) \end{aligned} \quad (18)$$

where S is the stiffness.

The work done thus depends upon the maximum curvature, ρ_{\max} . In each part of Eq. 18, the integral is the total work, derived in Eqs. (10) and (11), while the second term is the stored elastic energy.

It is interesting to note that the stiffness, S , changes when paper undergoes plastic deformation (bending or unbending) [17]. In order to reduce errors in the calculation of the bending work, W_b , we used S_b as the stiffness before bending produces significant plastic deformation, and S_{ub} for the rest. We use S_{ub} for the entire calculation of the unbending work.

The numerical solution of Eq. (18) is given in Eq. (G) of the Appendix. Fig. 3.8 shows the variation of the calculated irreversible work as a function of the maximum curvature ρ_{\max} for bending (curve a) and for unbending (curve b). In both cases, we observe that for small values of ρ_{\max} , W_b and W_{ub} are zero. This means that the paper undergoes elastic deformation in this range of curvatures. For the inelastic deformation, the work expended is an approximately linear function of the maximum curvature ρ_{\max} . They can be written as $W_b = \alpha_b \rho_{\max} + \beta_b$ and $W_{ub} = \alpha_{ub} \rho_{\max} + \beta_{ub}$. Thus, the work of

deformation, W_d , the sum of W_b and W_{ub} , will be an approximately linear function of ρ_{max} and can also be written as $W_d = \alpha_d \rho_{max} + \beta_d$. The values of the different slopes and intercepts are presented in Table 3.1.

Table 3.1: Calculated values of the slopes and intercepts of W_b , W_{ub} and W_d in their inelastic range when they are expressed as a function of ρ_{max} , for $30 \text{ g}\cdot\text{m}^{-2}$ basis weight detached strips.

Slopes $\cdot 10^{-3}$, N			Intercepts, $\text{N}\cdot\text{m}^{-1}$		
α_b	α_{ub}	$\alpha_d = \alpha_b + \alpha_{ub}$	β_b	β_{ub}	$\beta_d = \beta_b + \beta_{ub}$
$4,87 \pm 1,23$	$2,03 \pm 0,18$	$6,9 \pm 1,4$	$-0,58 \pm 0,1$	$-2,2 \pm 0,9$	$-2,78 \pm 1$

3.5. DETERMINATION OF THE WORK OF DETACHMENT

The separation of W_p into W_d and W_a may be accomplished by determining the experimental value of ρ_{max} during peeling and calculating the work of deformation, W_d . This separation may be effected using two approaches. First, we determine the values of W_b and W_{ub} corresponding to the measured value of ρ_{max} . Their sum is the work of deformation, W_d , dissipated during bending propagation, and the difference between W_p and W_d is the work of detachment W_a . The results are presented in Table 3.2, which gives the work of peeling supplied by the machine, W_p , the maximum curvature, ρ_{max} , and the

corresponding calculated value of W_d , and W_a obtained for various peel angles, θ .

It may be seen that ρ_{\max} increases with θ , leading automatically to an increase of W_d . In contrast, W_a does not exhibit any significant variation with ρ_{\max} or with θ . Consequently, we may conclude that the increase of W_p is due exclusively to the increase of W_d , and that W_a is independent of θ .

Table 3.2: Experimental values of work of peeling, W_p , and curvature, ρ_{\max} , with the corresponding calculated value of work of deformation, W_d , and work of detachment, W_a , for 30/30 g m⁻² laminated handsheet.

Angle, θ	90°	110°	130°	150°	170°
$W_p = \frac{P}{b} (1 - \cos\theta), \text{ N}\cdot\text{mm}^{-1}$	0,029	0,034	0,044	0,055	0,080
$\rho_{\max}, \text{ mm}^{-1}$	1,77	2,14	2,93	4,34	7,68
$W_d = W_b + W_{ub}, \text{ N}\cdot\text{mm}^{-1}$	0,011	0,015	0,022	0,033	0,060
$W_a = W_p - W_d, \text{ N}\cdot\text{mm}^{-1}$	0,018	0,019	0,022	0,021	0,020

The second approach may be seen as an application of the conclusion of the first approach. Since W_a is independent of ρ_{\max} but W_d is a linear function of ρ_{\max} , as shown in the previous section, W_p must also be a linear function of ρ_{\max} which may be written as $W_p = W_a + \alpha_d \rho_{\max} + \beta_d$. This can be verified experimentally.

Data showing the variation of W_p with ρ_{\max} for laminated handsheet 30/30 $\text{g}\cdot\text{m}^{-2}$ are plotted in Fig. 3.9. A straight line gives an acceptable fit to the experimental results. Thus, from the fitting, W_p can be written as $W_p = \alpha_p \rho_{\max} + \beta_p$. We will obtain α_p and β_p as the experimental slope and intercept respectively. Comparing the experimental and calculated results is equivalent to comparing the slopes, α_p and α_d , and to comparing the intercepts β_p and $W_a + \beta_d$, as shown in Table 3.3.

Table 3.3. Comparison between experimental and calculated slopes (α_p and α_d) and intercepts (β_p and $W_a + \beta_d$) in peel work, W_p , vs ρ_{\max} for laminated handsheet 30/30 $\text{g}\cdot\text{m}^{-2}$.

Slopes, N		Intercepts, $\text{N}\cdot\text{m}^{-1}$			
$\alpha_p \cdot 10^{-3}$	$\alpha_d \cdot 10^{-3}$	β_p	β_d	W_a	$(\beta_d + W_a)$
8,31±0,4	6,9±1,4	18,14±2,3	-2,78±0,5	20,8±2	18,02±2,5

From these results we see that the experimental slope, α_p , is equal to α_d within experimental error. The intercept, β_d , is small compared to β_p , or to the calculated detachment work, W_a , which are of the same order. This means that the contribution of

β_d to β_p in Fig. 3.9 may be neglected. Consequently, β_p may be taken as the detachment work, W_a , without significant error. Thus, we may determine W_a without having to measure the stress-strain properties, and without calculating W_d , as long as we determine W_p and ρ_{\max} as a function of θ .

3.6. CONCLUSIONS

Two consistent methods for separating peel work into work of deformation and work of detachment have been developed. We first presented a method for the determination of the deformation moment and work for paper. It is based on the true stress-strain curves for paper in tension and compression.

Using this, we have shown that the increase of the total work with peel angle, θ , is exclusively due to the work of deformation. The work of detachment, resulting from the difference between the work of peeling and work of deformation, is a constant, independent of θ . We then demonstrated a linear relation between work of deformation and maximum curvature. The work of detachment W_a may then be obtained from the extrapolation of $W_p(\rho_{\max})$ to $\rho_{\max}=0$, in good agreement with the detailed calculation:

The second method works because W_a is independent of θ and of ρ_{\max} . In subsequent articles, we shall demonstrate [18] that W_a is also independent both of peel speed and basis weight. We shall also present a simplified approach [19] which permits the determination of W_a , in agreement with the methods presented here, from peel test results alone, without measuring ρ_{\max} .

3.7. ACKNOWLEDGMENT

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Appendix

Bending equation

Stress equilibrium is expressed by Eq. (15):

$$\int_0^{h_t} \sigma_t(z) dz = \int_0^{h_c} \sigma_c(z) dz, \quad (\text{A})$$

where σ_n and h_n are the absolute values of stress and thickness in tension ($n=t$) and compression ($n=c$). For a beam subjected to bending, we obtain from Eq. (6):

$$z=R\varepsilon \quad \text{and} \quad dz=Rd\varepsilon,$$

where R is the radius of curvature of the beam at the neutral surface and ε is the strain at a distance z from the neutral surface.

We have defined ε_t and ε_c as the strains in tension and compression, at the surfaces Eq.

(A) then becomes

$$\int_0^{\varepsilon_t} \sigma_t d\varepsilon = \int_0^{\varepsilon_c} \sigma_c d\varepsilon. \quad (\text{B})$$

A numerical solution of Eq. (B) permits us to obtain the positions of the neutral surfaces, i.e., the value of h_t and h_c , when the stress-strain data are known, as shown in Fig. 3.3.

The trapezoidal formula for the numerical solution of integrals applied to Eq. (B) may be expressed as

$$\sum_{i=0}^{T-1} \left(\frac{\sigma_{i+1} + \sigma_i}{2} \right) (\epsilon_{i+1} - \epsilon_i) = \sum_{j=0}^{C-1} \left(\frac{\sigma_{j+1} + \sigma_j}{2} \right) (\epsilon_{j+1} - \epsilon_j) \quad (\text{C})$$

In Eq. (C), σ_n and ϵ_n are the stress and strain corresponding to the point n .

To solve Eq. (C), a value of ϵ_T (T represents the last term in the left hand-side of Eq. (C)) is selected as the maximum tensile strain. An iterative procedure is then applied until Eq. (C) is satisfied. Finally, the radius of curvature and the thicknesses h_t and h_c are obtained by solving Eqs. (6) and (16). These give:

$$h_t = \frac{\epsilon_T}{\epsilon_T + \epsilon_C} \quad ; \quad h_c = \frac{\epsilon_C}{\epsilon_T + \epsilon_C} \quad ; \quad R = \frac{h}{\epsilon_T + \epsilon_C} \quad (\text{D})$$

The values of σ_t and σ_c are determined from experiment (Fig. 5). The same procedure is used for unbending, except that we use Eq. (8) rather than Eq. (6).

The sets of values of stress and strain in compression and in tension are stored in matrix rows as $(\epsilon_{t,i}, \sigma_{t,i}, \epsilon_{c,i}, \sigma_{c,i})$, where i represents the row number, and t and c refer to the tension and compression mode, respectively.

Therefore, the moment can be calculated by the numerical solution of Eq. (17)

$$M_N = \frac{1}{2\rho_N^2} \sum_{i=0}^{N-1} ((\sigma_{t,i+1}\epsilon_{t,i+1} + \sigma_{t,i}\epsilon_{t,i})(\epsilon_{t,i+1} - \epsilon_{t,i}) + (\sigma_{c,i+1}\epsilon_{c,i+1} + \sigma_{c,i}\epsilon_{c,i})(\epsilon_{c,i+1} - \epsilon_{c,i})) \quad (\text{E})$$

where $\rho_N = \frac{\epsilon_{t,N} + \epsilon_{c,N}}{h}$ is the curvature at which the moment is measured.

Finally, the work of bending and unbending at each curvature may be calculated by the numerical solution of Eq. (18):

$$W_N = \sum_{i=0}^{N-1} \left(\frac{M_{i+1} + M_i}{2} (\rho_{i+1} - \rho_i) \right) - \left(\frac{M_N^2}{2S} \right) \quad (\text{G})$$

where ρ_N represents the maximum curvature of the bent portion of the detached layer.

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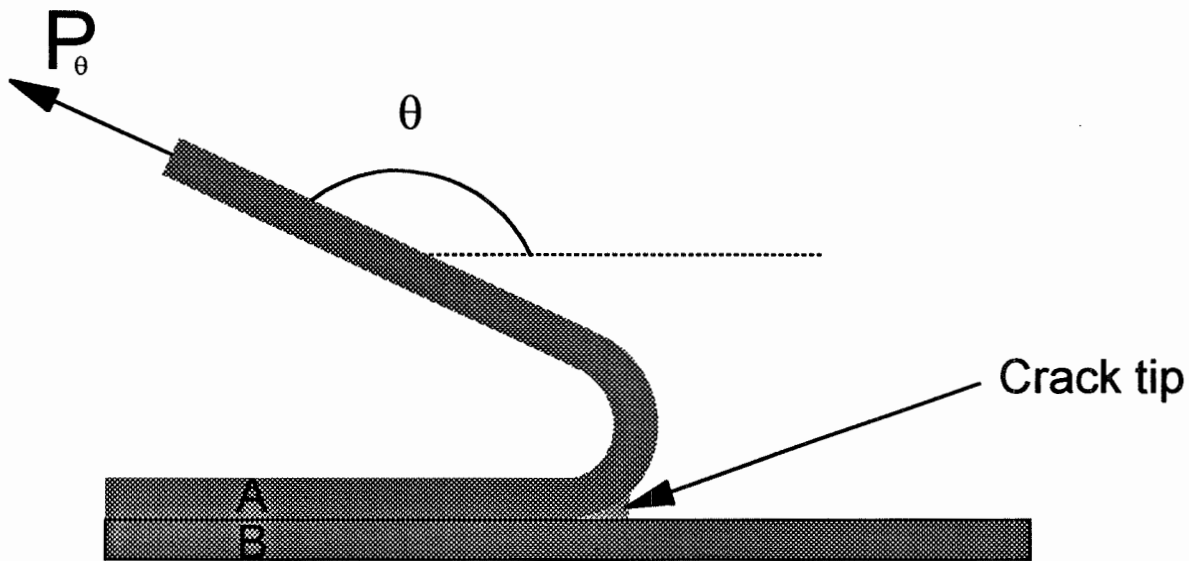


Figure 3.1: Configuration of a delaminated sample in a peel test.

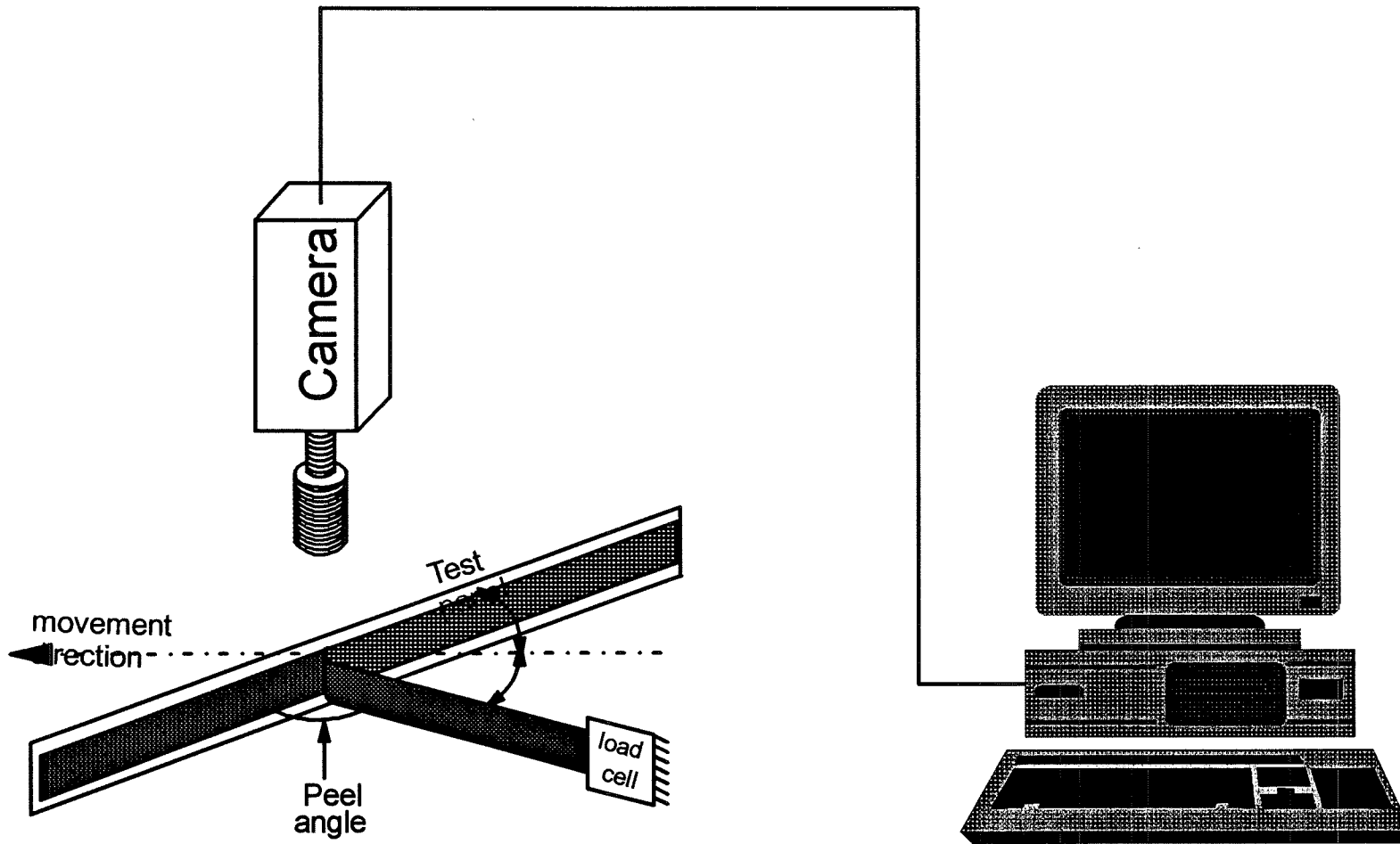


Figure 3.2: Schematic representation of the peel tester equipped with a video camera.

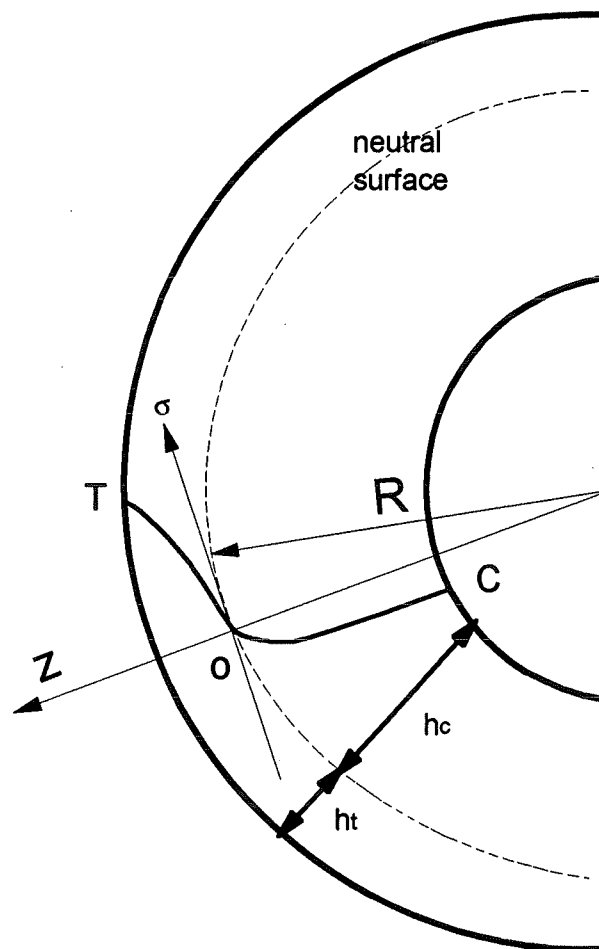


Figure 3.3: Variation of stress in the z -direction during the bending of paper. T and C represent the values of tensile and compressive stress at the surfaces. R is the radius; h_t and h_c are the thicknesses of the stretched and compressed sides respectively.

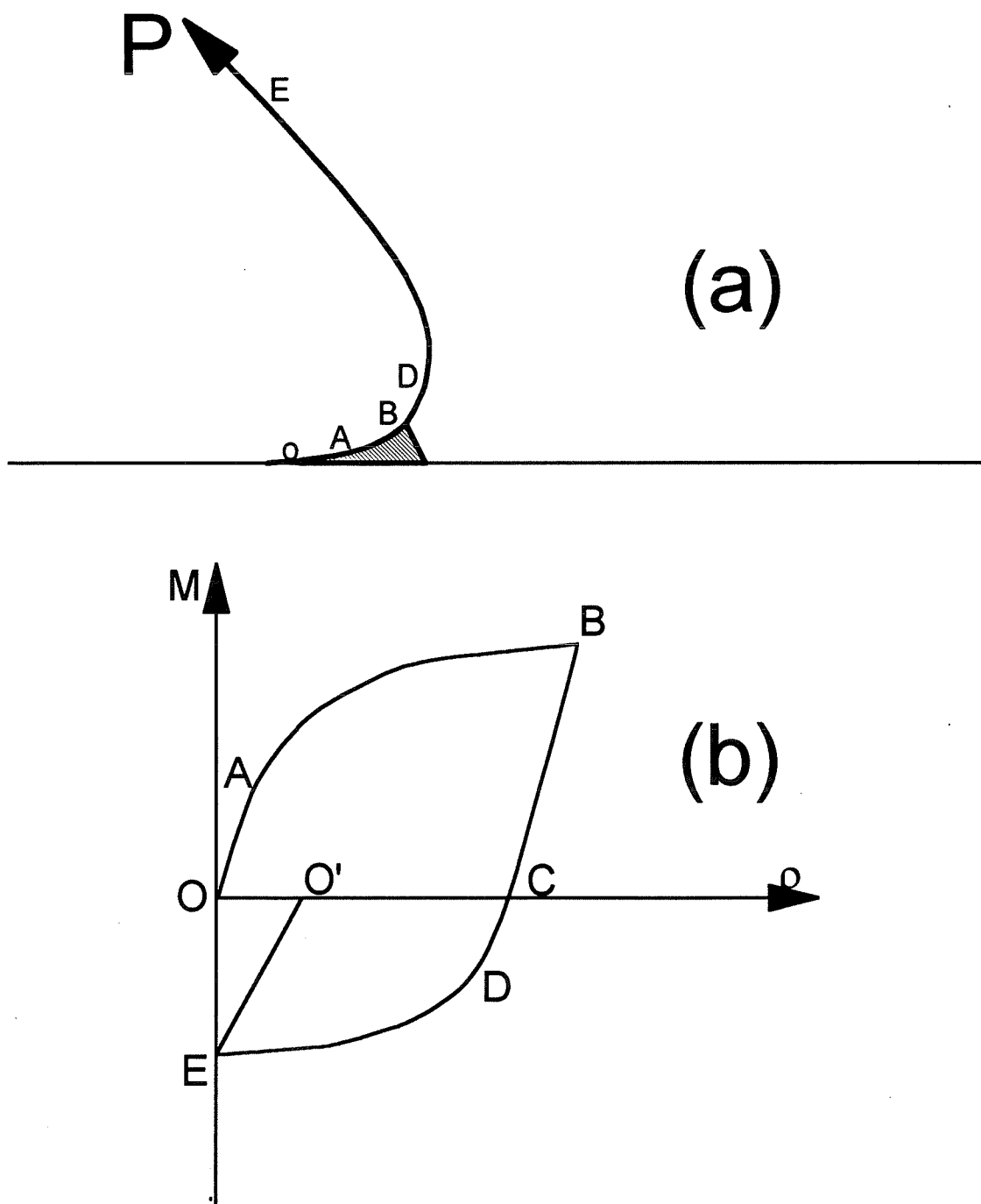


Figure 3.4: Correspondence between the moment/curvature curve and the state of the sheet during peeling: (a) peel shape, (b) hysteresis of the bending moment as a function of the curvature.

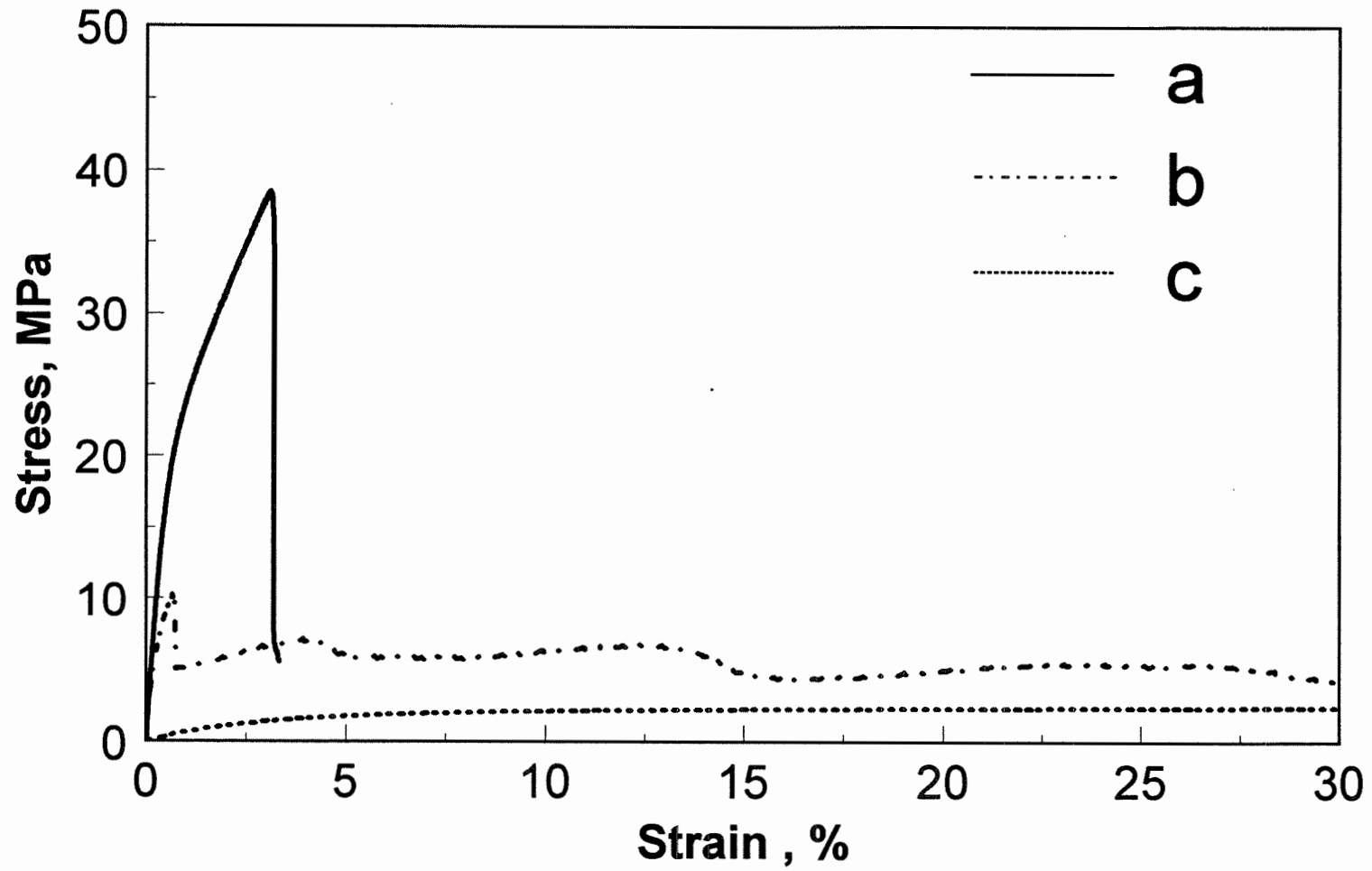


Figure 3.5: Stress-strain curves of paper during (a) stretching of the virgin sample, (b) compression of the virgin sample and (c) stretching of a previously compressed sample.

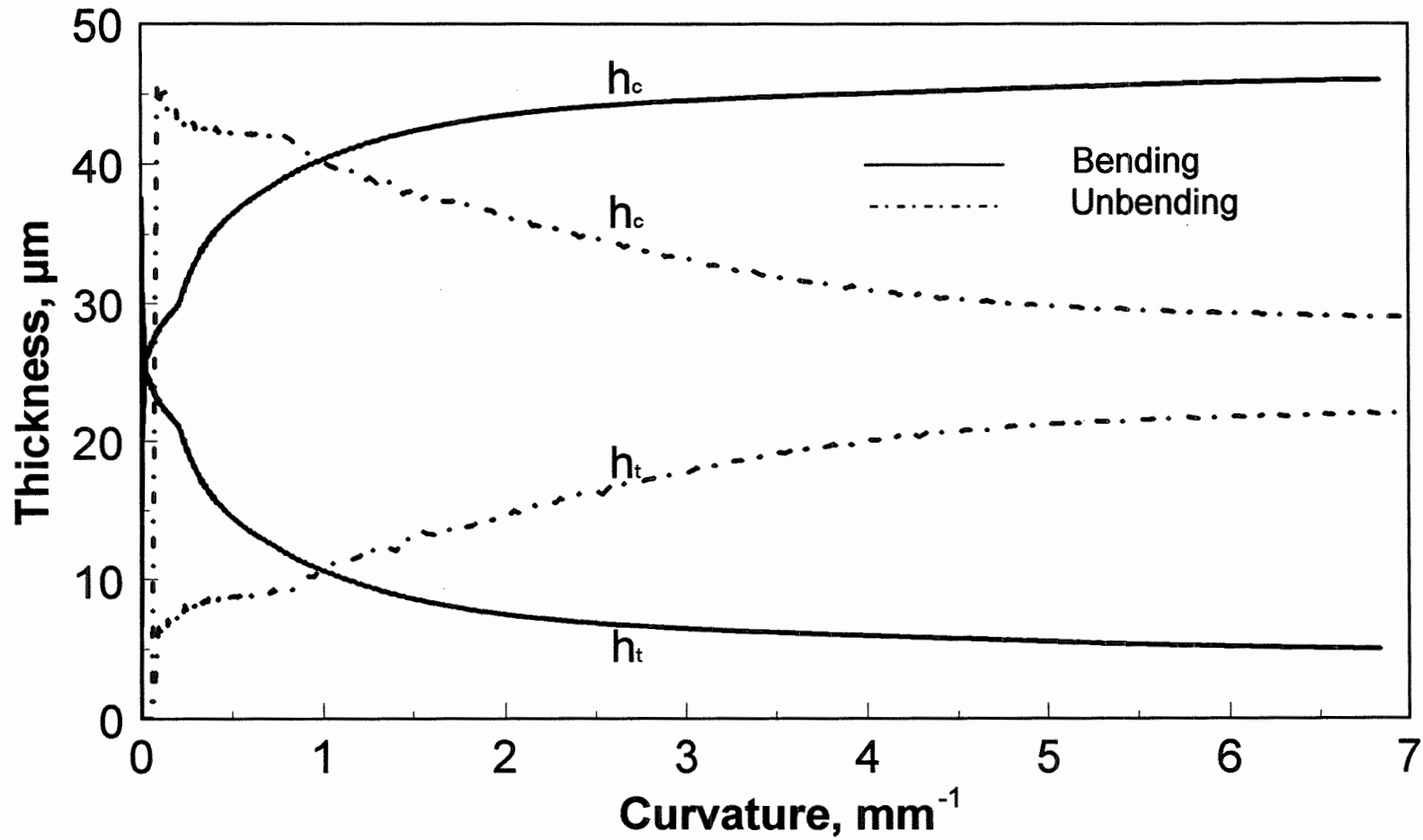


Figure 3.6: Calculated thicknesses of stretched (h_t) and compressed (h_c) regions during bending (solid line) and unbending (dashed line) of a layer 50 μm thick, corresponding to a 30 g/m^2 basis weight.

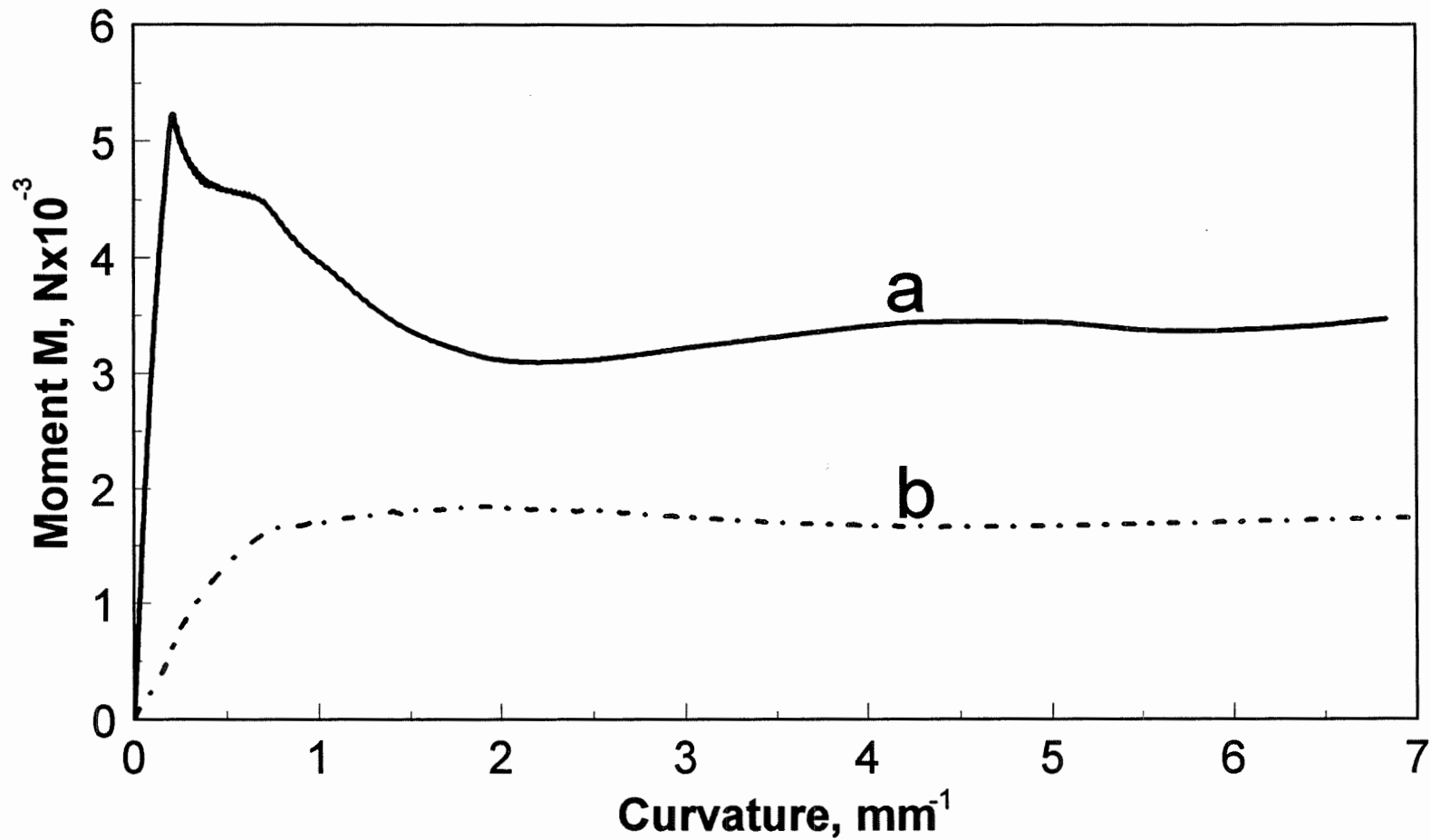


Figure 3.7: Bending(a) and unbending (b) moments calculated using Eq. E for a layer 50 μm thick, corresponding to a 30 g/m^2 basis weight. The abscissa, represents ρ for M_b and ρ' for M_{ub} .

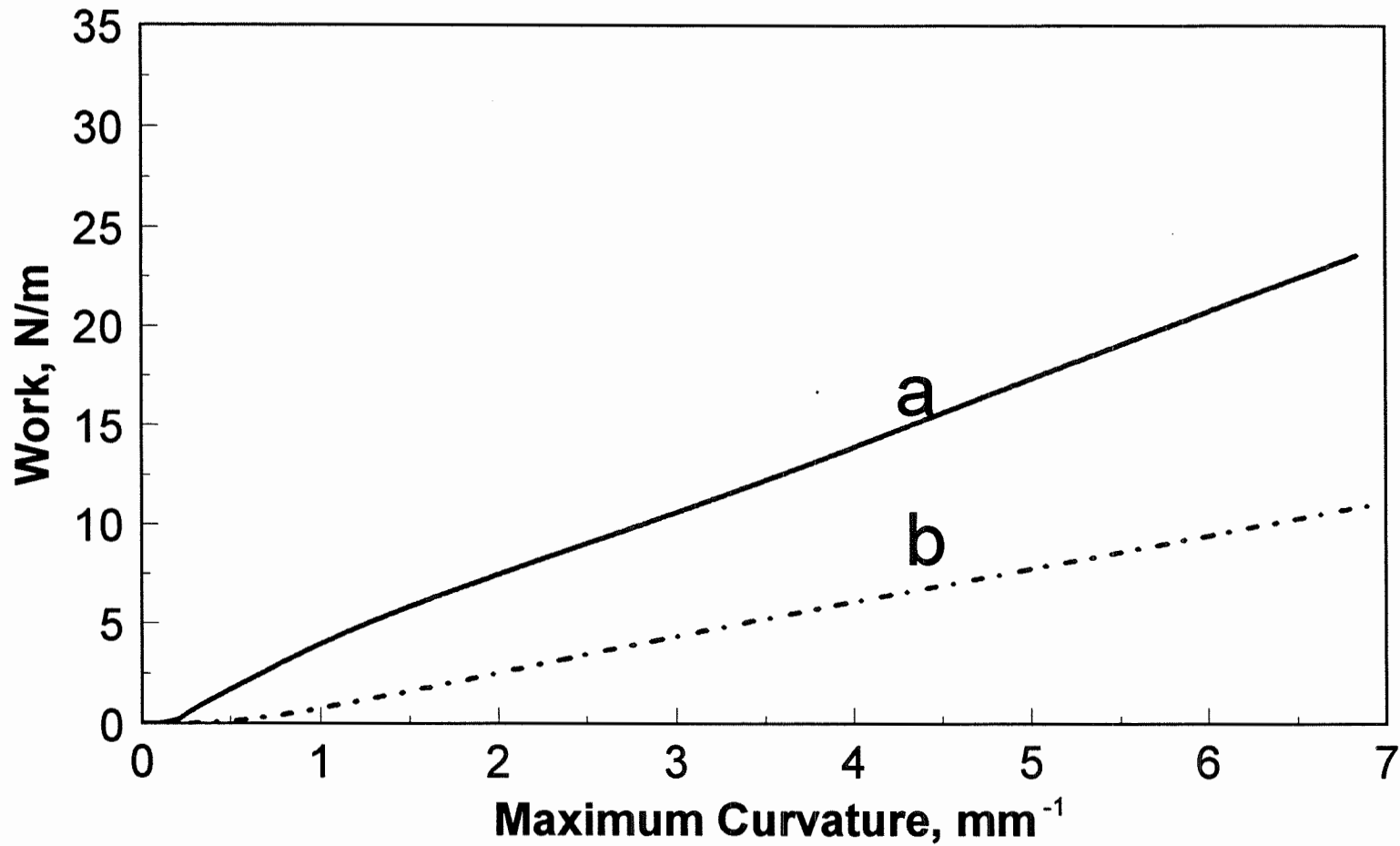


Figure 3.8: Bending (a) and unbending (b) work calculated using Eq. G for a layer $50\mu\text{m}$ thick, corresponding to a 30 g/m^2 basis weight. The curvature abscissa represents the maximum curvature for bending, and the initial curvature for unbending.

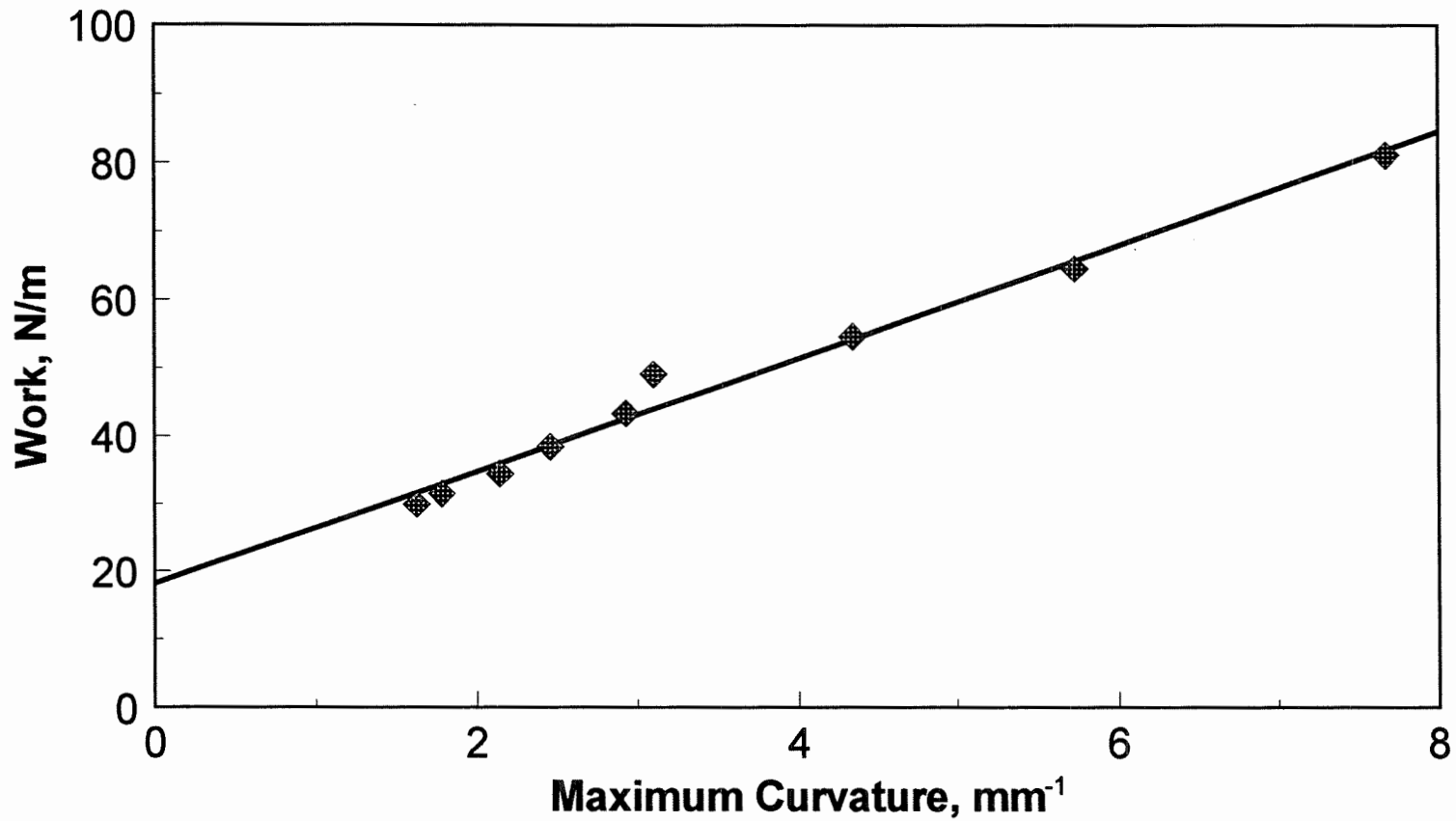


Figure 3.9: Variation of work of peeling with the maximum curvature for laminated handsheets of 30/30 g/m² basis weight.

CHAPITRE 4

Angle-Dependent Delamination of Paper.**Part III: Effect of Work of Detachment on Work of Peeling**

Angle-Dependent Delamination of Paper.

Part III: Effect of Work of Detachment on Work of Peeling

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ABSTRACT

In this study, we describe the effect of the basis weight of paper on W_a and the mutual relation between W_a and the bending curvature, ρ_{\max} . We show that the basis weight does not modify W_a but that W_a and stiffness both affect ρ_{\max} and, therefore, change W_d . The effect of W_a on ρ_{\max} was shown to produce an amplification phenomenon which explains the high sensitivity of the peel test and the large, experimentally observed, fluctuations of the peel force.

4.1. INTRODUCTION

The peel test has been used to investigate the mechanical properties of paper in the thickness direction. It has been assumed [1, 2] that the total peel work, W_p , went into creating a new surface, by splitting a sheet into two layers. Thus, W_p has been considered [1,2] to be a measure of z-bond toughness. Also, a simple relationship between W_p (called the « the mechanical work of adhesion ») and the thermodynamic surface tension has been proposed [3]. However, we have shown [4] that W_p is the sum of two terms: the detachment work, W_a , expended in separating two layers, and the deformation work, W_d , expended in bending the detached upper layer. It was shown [5] that W_d may be considerably larger than W_a . The terms employed in this study are defined in Table 4.1 and in previous publications [4,5]

Table 4.1. Nomenclature and definition of principal terms

Symbol	Nomenclature	Definition and units
W_p	work of Peeling	Total work per unit area supplied by peel tester, $N \cdot m^{-1}$ or $J \cdot m^{-2}$.
W_a	work of Detachment	Work needed to separate top from bottom layer during peeling, $N \cdot m^{-1}$ or $J \cdot m^{-2}$.
W_d	work of Deformation	Work spent during bending propagation in the detached top layer, $N \cdot m^{-1}$ or $J \cdot m^{-2}$.
ρ_{max}	Maximum curvature	Maximum curvature reached, during peeling, in the bent part of the top layer, m^{-1} .
---	Bond toughness	Work per unit bonded area required to break bonds, $N \cdot m^{-1}$ or $J \cdot m^{-2}$.

In general, detachment may involve two modes of fibre deformation and bond-breaking: cleavage, or mode I, and shear, or mode II. Likewise, bending of a sheet involves both compression and tension on different faces of the delaminated upper layer. In-plane tensile tests may lead to breakage in mode II [6]. In-plane compression may lead to failure in both modes I and II [7-9]. Both of these are different from z-bond toughness which involves only breaking in mode I. Work of peeling at small and large angles will involve both modes, as well. However, work of detachment, W_a , should reflect z-bond toughness properties when the measurements are done at large peel angles ($90^\circ - 180^\circ$) [10]. Here, W_a is obtained principally in mode I (cleavage), since most bond areas are roughly parallel to the sheet plane. Therefore it is essential to demonstrate that W_a is a pure function of fibre-fibre bond energy, that it is not affected by the peeling process. Hence, it is important to examine the effects of the thickness and stress concentration upon W_a , and the effect of W_a upon the curvature of the delaminated part, and on the work of deformation W_d .

The effect of stress concentration on failure strength is well established for many materials. The strength of any material is limited by the presence of flaws, which distort the stress field in the sample and cause failure by initiation and propagation of a crack when a critical stress level is reached. But the stress concentration also depends upon the test method used, as has been reported previously [8,11,12]. The effect of stress concentration can be revealed by some parameters which depend upon the type of test

[11]. For example, in the z-direction tensile test, the basis weight of the samples was varied in order to investigate the effect of stress concentration.

For peel test, Van Den Akker [11] suggested that varying the stiffness of the detached strip should reveal the effect of stress concentration. The increase of maximum curvature, ρ_{\max} , due to a decrease in stiffness, should lead to a reduction of the detachment zone size, and to an intensification of the stress; thus the work of detachment will decrease.

The effect of W_a upon ρ_{\max} and W_d was reported by Mantel and Descaves [13] for metal/polymer/metal sheet sandwiches. They found that as W_a and W_d increase the bending radius will decrease with an increase in peel speed. They explain their results as the effect of speed upon W_a which in turn affects the bending curvature. However, due to the complex effect of speed on paper (due to the rheological properties in bending [4,5]), their method can not be directly applied to peeling of paper.

In this study, we shall describe the effect of basis weight of paper on W_a and the mutual relation between W_a and bending curvature, ρ_{\max} . We shall show that basis weight does not modify W_a , but that W_a and stiffness can both affect ρ_{\max} , and therefore can change W_d . The effect of W_a on ρ_{\max} will be shown to produce an amplification phenomenon which can explain the high sensitivity of the peel test and the large, experimentally observed, fluctuations of peel force.

4.2. EXPERIMENTAL

4.2.1. Sample preparation

Handsheets were made from bleached Kraft pulp beaten to 500 rev. in a PFI mill. The samples were prepared by lamination of two handsheets under wet pressing pressure at 420 kPa. The samples used to investigate the effect of bending curvature and of corona treatment on peel results had basis weight 30/30 g·m⁻² (a/b means a g·m⁻² for the top layer and b g·m⁻² for the bottom layer). In order to characterize the effect of the thickness of the bottom layer, samples varying in basis weight (from 30/30 g m⁻² to 30/80 g·m⁻²) were prepared.

4.2.2. Corona treatment

The corona treatment apparatus is shown in Fig. 4.1. It has a moving grounded plate electrode made of aluminum, driven by an electric motor at a controlled speed. The upper quartz covered electrode was supplied by a high voltage AC generator [14]. The gap between the two electrodes is 2.5mm. For all corona treatments, the voltage was fixed at 18 kV, the measured current was 10 mA. Treatment energy was adjusted by using different plate speeds. The wet handsheets were fixed on the plate and treated by moving them through the corona discharge zone.

The degree of treatment is given by:

$$\text{degree of treatment} = \frac{W}{V \cdot L} \quad [\text{J} \cdot \text{m}^{-2}]$$

where W is the electric power (W), V is the speed ($\text{m}\cdot\text{s}^{-1}$), and L is the length of the upper electrode, ($L= 0.238$ m). The corona treatment was carried out in air, and the treated surfaces of two handsheets were laminated following the procedure described above. The peel test and sample preparation have been described in detail elsewhere [4,5]. In this study, the peel speed was fixed at 2.54 $\text{cm}\cdot\text{min}^{-1}$.

4.3. RESULTS and DISCUSSION

In Fig. 4.2 we show the video image of the detachment zone of a sample during peeling. In the zone just behind the crack, fibers are deformed but still bridge the top and the bottom layers. The observed deformation is progressive and reaches its maximum at the crack tip, where the maximum curvature of the top layer, ρ_{max} , is observed.

Paper is a ductile material [15]. Hence, the observed z-deformation is inelastic, and the work of detachment is the sum of the energy consumed for the z-deformation and that spent to create a new surface [15,16]. Therefore, to characterize W_a , it is of major importance to examine the depth affected by the z-deformation process. If W_a is characteristic of the behavior at the interface, W_p should be independent of sample thickness. If the value of W_a is a function of sample thickness, W_p will also vary with the sample basis weight and will be affected by the flaws present in the bulk of the sample.

4.3.1. Effect of thickness of the bottom layer.

Since the bottom layer of a laminated sample is always kept flat during the delamination, the only process which may be affected by this layer and by its thickness is the detachment. Hence, to determine how much of the bulk of the paper is affected by z-deformation, work of peeling measurements were performed for handsheets of various bottom thicknesses, of basis weight ranging from 30 to 80 g·m⁻².

Fig. 4.3a shows the measured peel work for the various bottom layer thicknesses, at three different peel angles. There is no significant effect of bottom layer thickness. Furthermore, the increase of the sample thickness due to peeling, Δt (the difference between the thickness after, t_a , and before, t_b , delamination, $\Delta t = t_a - t_b$), does not change with the thickness of the bottom layer, as shown in Fig 4.3b. Thus, only the interface region of the bottom layer is affected by the z-deformation. Consequently, W_a and W_p cannot be affected by flaws in the bulk of the sample, except those at the interface of the detachment zone.

It should be noted here that the z-deformation behavior described above is specific to laminated handsheets. In contrast, in the delamination of single handsheets (in the same range of thicknesses as used in this work) the whole sample thickness is affected by the z-deformation. For this case it has been shown that the work of peeling and Δt increase with the paper basis weight [17]. The difference arises, of course, from the

entanglement of the fibres in single handsheets, as opposed to the well-defined delamination surface in our case.

4.3.2. Effect of bending curvature on work of detachment.

For the peel test, the increase of the curvature in the detachment zone has been described as an intensifier of the stress [11]. Thus, it was predicted that the work of detachment decreases with the increase of the curvature. To verify this effect, we changed the radius in the detachment zone without affecting the dissipation properties of top layer in bending. This was done by applying an adhesive tape to the top layer.

The use of pressure sensitive tape to reinforce the layer subjected to peeling has been reported previously [1,2,17]. The authors assumed that the peeling process was perfectly elastic. Therefore no consideration was given to a possible effect of the tape on the results. However, our previous work [4,5] has shown that most of the energy dissipated in the peeling of laminated paper goes into flexion of the top layer. Therefore, a significant effect of the tape on the conditions of the peeling process and on the results obtained is to be expected.

First, we wished to determine whether the tape contributes to the deformation work. We therefore examined the behavior of tape during peeling. Then, we compared the results obtained for paper samples with and without tape on their top layers (called samples of types A and B, respectively).

In the first such experiment, pressure sensitive tape was attached to a flat metal plate previously cleaned with alcohol. The resulting peel force was four times higher than that measured for paper samples in the same conditions. The detachment was clearly interfacial in character, that is the separation took place at the interface between the adhesive and the plate [18,19]. This shows that the adhesive of the tape is significantly stronger than the bond strength in paper, and did not take part in the delamination of paper samples at the speeds studied. Consequently, the adhesive at the paper-tape interface is also negligibly affected by the deformation during the bending.

In a second experiment, various amounts of an inert powder were applied uniformly to the tape in order to weaken the strength of its adhesive, before sticking it to the metal plate. The resulting peel force could be adjusted by the amount of powder used to obtain a peel force close to that measured for the paper samples. It was observed that the tape did not undergo plastic or viscoelastic deformation in this case. This means that under the conditions of paper delamination, the tape exhibited only elastic behavior.

The elastic role of tape in reinforced paper samples can be understood by comparing the results obtained for samples with (A) and without (B) tape. Both types of samples were prepared from the same laminated handsheet ($30/30 \text{ g}\cdot\text{m}^{-2}$). Fig. 4.4 shows the variation of the work of peeling with applied peel angle. Fig. 4.5 shows the maximum curvature, measured in the bent region. It may be seen that application of the adhesive tape to the top layer reduces the work of peeling and the maximum curvature for all applied peeling angles. We have shown in ref. [5] that the most important contribution to work of peeling

of laminated paper is the deformation work spent in bending and unbending the top layer. This work is directly related to the curvature of this layer.

Fig. 4.6 shows W_p as a function ρ_{\max} for both samples A and B. All the results fit the same line, showing that work of deformation was due to the bending of paper only, and not of the tape. The tape increased the stiffness of the strip subjected to peeling. Consequently, the increase in radius in the bent part causes a decrease of work of deformation, W_d . We may say that, in general, the deformation of the tape during peeling is purely elastic when it is used to reinforce the top layer of paper samples. The paper was the only element which underwent irreversible deformation.

The work of detachment W_a was determined for samples of types A and B, using the method developed previously, which makes use of the measured mechanical properties [5]. At each peeling angle the values of W_a calculated for samples A and B were similar, as shown in Table 4.2. This is another way of treating the data presented in Fig. 4.6, where the results for both samples (A and B) fit the same line. The work of detachment (the intercept of the straight line), $19,9 \text{ N}\cdot\text{m}^{-1}$, is in agreement, within experimental error, with the value of W_a reported in Table 4.2.

Table 4.2. Comparison between results obtain on both samples, A and B

θ	Sample A, with adhesive tape.				Sample B, without adhesive tape.			
	P N·m ⁻¹	W _p N·m ⁻¹	ρ_{\max} mm ⁻¹	W _a N·m ⁻¹	P N·m ⁻¹	W _p N·m ⁻¹	ρ_{\max} mm ⁻¹	W _a N·m ⁻¹
90°	25,7	25,7	1,02	20,0	29,7	29,7	1,63	19,2
130°	19,0	31,2	1,77	19,2	26,4	43,3	2,93	21,7
170°	25,7	51,1	3,90	21,3	40,9	81,2	7,67	20,0
W _a				20,3				20,3
Average								

We conclude that the work of detachment, W_a , is characteristic of the interface and is not affected by the radius of curvature of the detached layer. For all laminated papers investigated, stress intensification did not play a significant role in the detachment, contrary to the prediction of Van Den Akker [11].

4.3.3. Effect of detachment work upon bending curvature and deformation work.

We have shown that the work of detachment for laminated paper is characteristic of the delaminating interface and is independent of the bending curvature. We may say that work of deformation, W_d , does not affect W_a . However, it may be shown that W_a does affect the radius of the bent part of delaminated layer [13]. Stronger bonding at the interface leads to a

reduction of the bending radius during peeling, and to an increase of W_d , the energy expended in bending and unbending.

To study this effect, we used corona treatment to modify the bond strength at the interface of laminated handsheets. Such a treatment did not affect the bending dissipation properties of the laminated layers. Corona discharge has been found to enhance bond strength at the interface when applied to the paper surfaces in the wet state, before lamination [20].

Fig. 4.7 shows the dependence of work of peeling, W_p , on the degree of interface treatment, defined as corona treatment energy per unit area, for three different angles. These results are in agreement with those of Sakato et al [20]. To separate the effect of corona treatment on W_a and W_d , we have plotted W_p as a function of ρ_{max} , as shown in Fig. 4.8. As discussed above and in [5], W_p is a linear function of ρ_{max} . The intercept of the line is W_a , while the slope characterizes the dependence of W_d upon the radius. Samples with the same mechanical properties should show the same slopes.

It may be seen from Fig. 4.8 that the results for different degrees of treatment yield parallel straight lines. This means that the work of detachment, W_a , increased with the degree of treatment, while the deformation work was not affected. This is in agreement with reports in the literature [20,21] on the effect of corona on paper.

In order to show the effect of work of detachment upon the bending curvature (and on work of deformation), we present, in Fig. 4.9, the measured value of ρ_{max} as a function of the calculated values of W_a , for four different angles. As expected, ρ_{max} increased with W_a ,

showing that stronger bonding at the interface produced a reduction of the peel radius. This, in turn, produced an increase of the energy expended in bending.

These results show that flexion in the peeling process amplifies changes in W_a .

The relation between ρ_{\max} and W_a can be approximated by:

$$\rho_{\max} = \rho_0 + A_\theta W_a \approx A_\theta W_a \quad (1)$$

In Eq. (1) ρ_0 is the curvature at $W_a=0$, which represents essentially elastic flexion. Hence, ρ_0 is very small and may be neglected. The slope, A_θ , depends upon the rheological properties of the top layer and increases with the peel angle, as shown in Fig. 4.9. Since W_d is a linear function of ρ_{\max} , W_p will be given by:

$$W_p = [1 + \alpha_p A_\theta] W_a \quad (2)$$

where $(1 + \alpha_p A_\theta)$ is the amplification coefficient, and α_p is the slope of the curve $W_p = \alpha_p \rho_{\max} + W_a$ reported in ref. [5].

This observation explains the high sensitivity of the peel test compared to the ultrasonic test in the z-direction [2]. It also helps to explain the magnitude of the fluctuation of the peel force, as discussed below.

4.3.4. Fluctuation phenomena.

When paper is delaminated at a constant rate, the measured force, in the ideal case, should be constant. In practice, however, the peeling force is very noisy, as shown

in Fig. 4.10. The observed noise is due to fluctuations of interfacial bonding and to the distribution of mechanical properties of the detached layer.

Fig. 4.11a shows the average peel force, P , and noise amplitude, ΔP , vs θ , ΔP is defined as the standard deviation of P . In Fig. 4.11b, we show $\frac{\Delta P}{P}$ as a function θ . It may be seen that the curves of ΔP and of P show the same general shape, but that $\frac{\Delta P}{P}$ increases for higher values of peel angle. For a linear amplifier, which the measurement system is, the source and resulting relative errors should be the same and should not depend upon peel angle, as was observed in practice. Thus, the properties of the sample must also fluctuate.

In fact, variation of work of detachment (ΔW_a) results in a significantly higher variation of peel work (ΔW_p). This is due to the additional energy spent for bending at increased curvature [4,5]. The observed variation of work of peeling $\Delta W_p = \Delta W_a + \Delta W_d$ is significantly larger than ΔW_a alone. The relative change of work of peeling, which is equal to the relative change of peel force, can be then written as:

$$\frac{\Delta P}{P} = \frac{\Delta W_p}{W_p} = \frac{\Delta W_a}{W_a} + \frac{\Delta \alpha_p}{\frac{1}{A_\theta} + \alpha_p} + \frac{\Delta A_\theta}{A_\theta + \frac{1}{\alpha_p}} \quad (3)$$

The term $\frac{\Delta \alpha_p}{\frac{1}{A_\theta} + \alpha_p}$ is an increasing function of peel angle. It is responsible for the increase of $\Delta P/P$ with peel angle. This is in agreement with the results presented in Fig. 4.11.

Fluctuations of W_a due to local sample imperfections are amplified by the peeling process. This results in large fluctuations in peel force, causing the increase in noise, especially for higher values of peel angle.

4.4. CONCLUSIONS

We have demonstrated that the work of detachment, W_a , is characteristic of the interface of laminated sample and is independent on the thicknesses of upper and bottom layers and of bending radius which was predicted to be the source of the stress concentration. Hence, if we reduce the W_d , by increasing the stiffness of the top layer and using the appropriate peel angle, to be negligible in comparison to W_a , it may be the way to determine the mechanical properties of the fibre-fibre bond in z-directions, especially the z-bond toughness. In contrast, the bending radius and work of deformation are functions of the work of detachment, being dependent on the mechanical properties of top layer. Flexion thus amplifies the peel force. This explains the sensitivity of peel test to fluctuations in W_a and the large amplitude of the observed noise.

4.5. ACKNOWLEDGMENTS

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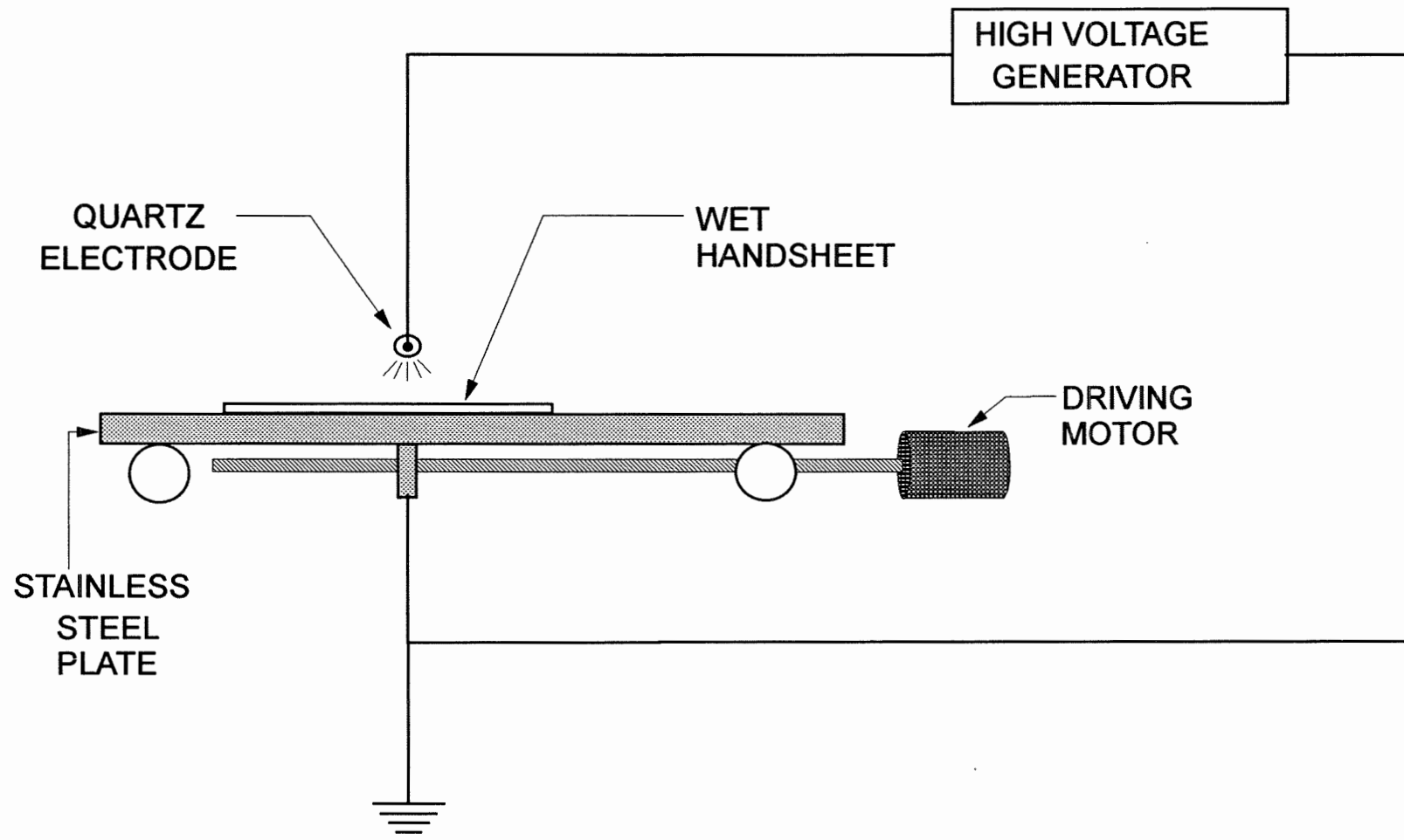


Figure 4.1. Diagram of corona treatment apparatus.

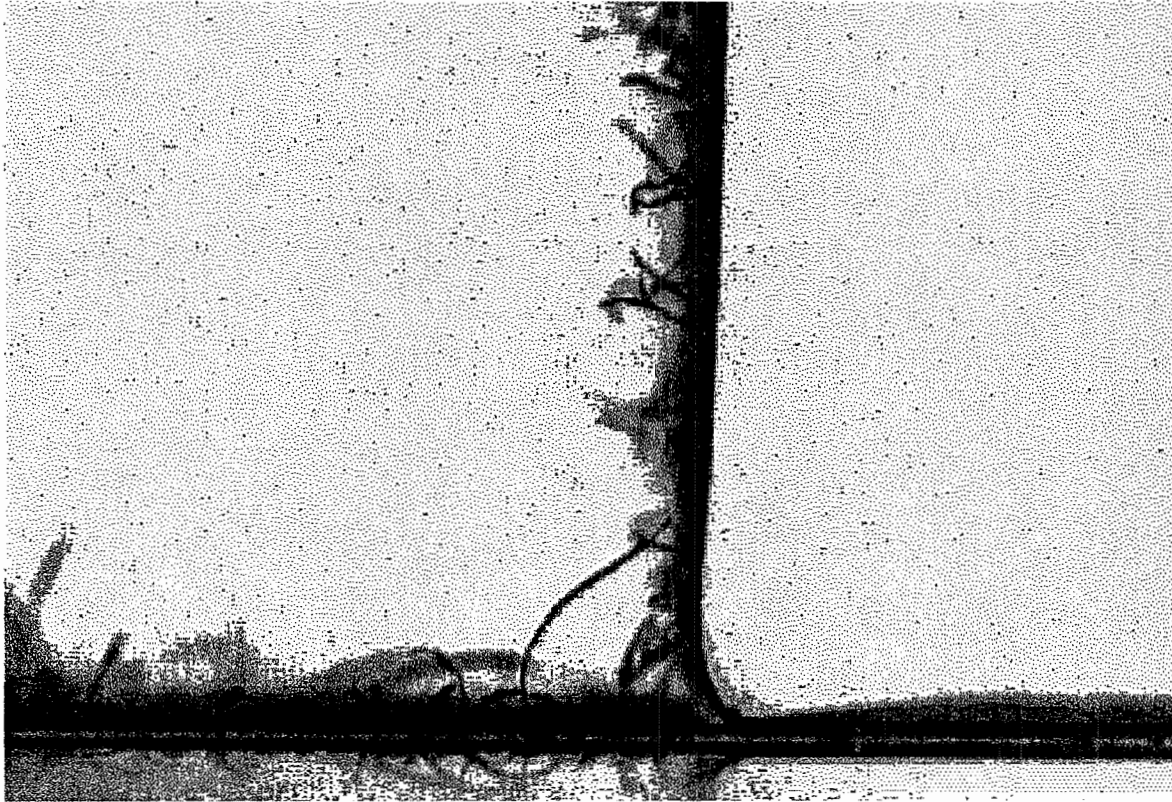


Figure 4.2. Image of the detachment zone taken by video camera.

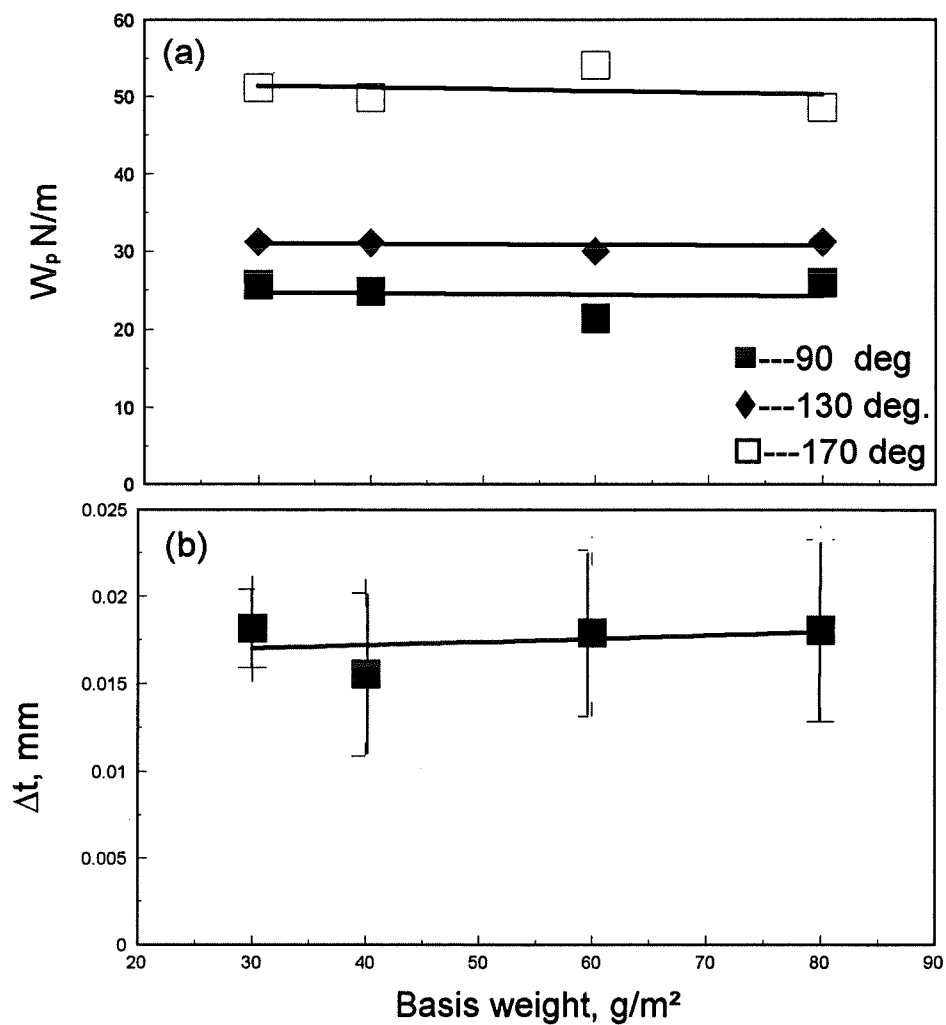


Figure 4.3. (a) Variation of work of peeling with the basis weight of bottom layer at three angle 90°, 130° and 170°, (b) The increase of the thickness, Δt , for samples peeled at 90°, as a function of the basis weight of the bottom layer.

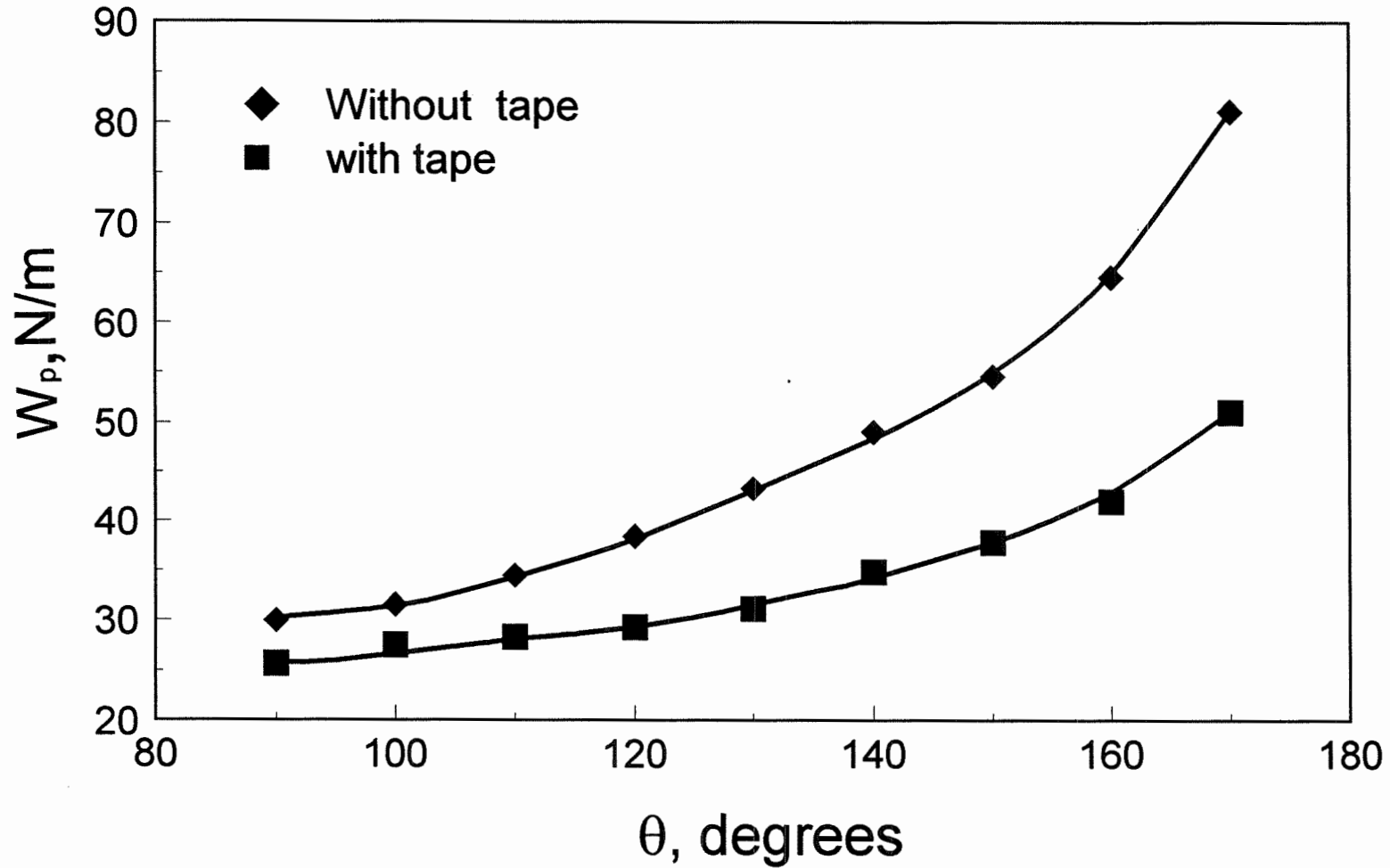


Figure 4.4. Variation of work of peeling, W_p , with q for samples with the top layer reinforced by adhesive tape (■) and without tape (◆).

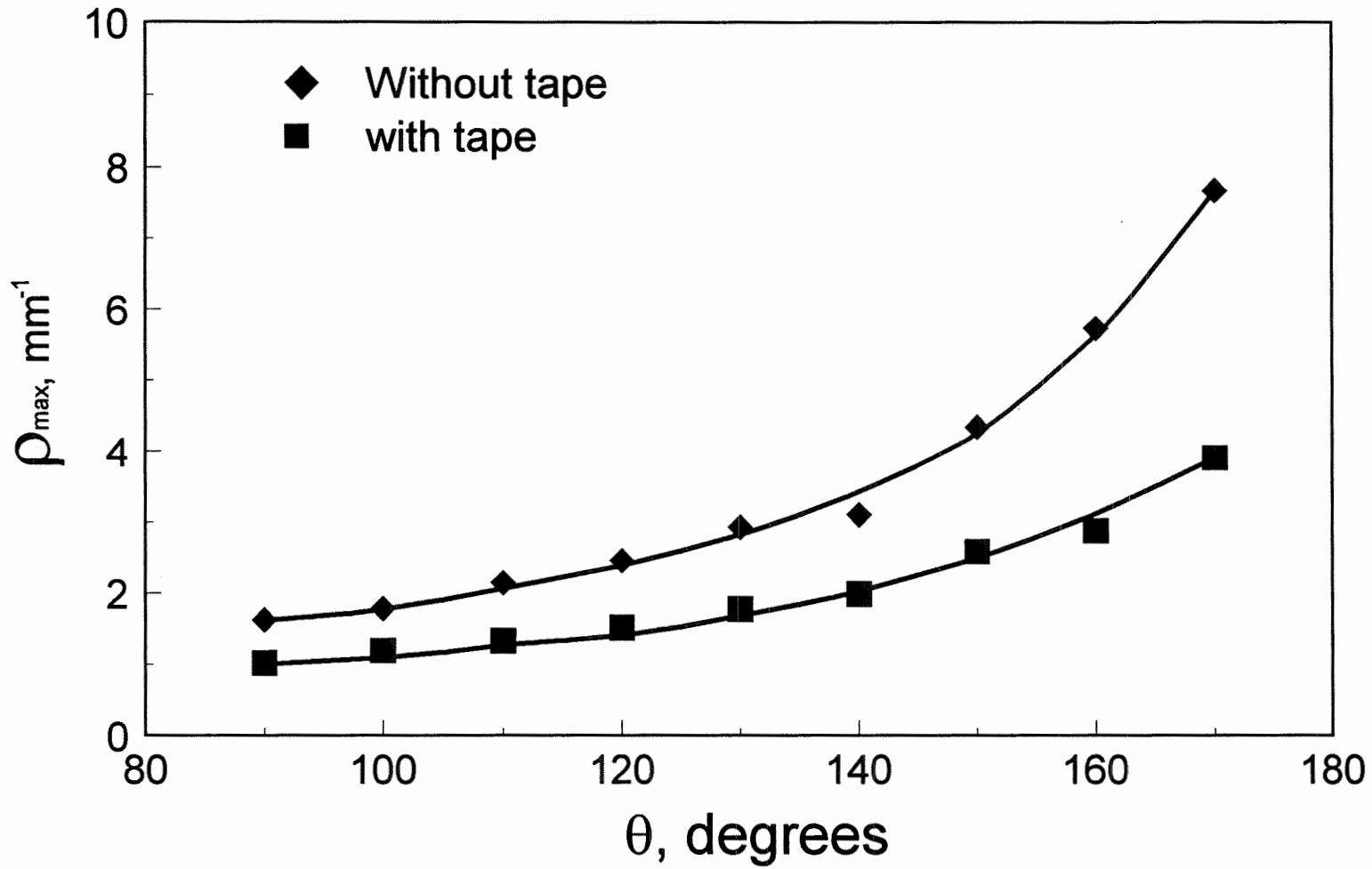


Figure 4.5. Variation of curvature, ρ_{\max} , with q for tope layer reinforced by adhesive tape (■) and without tape (◆).

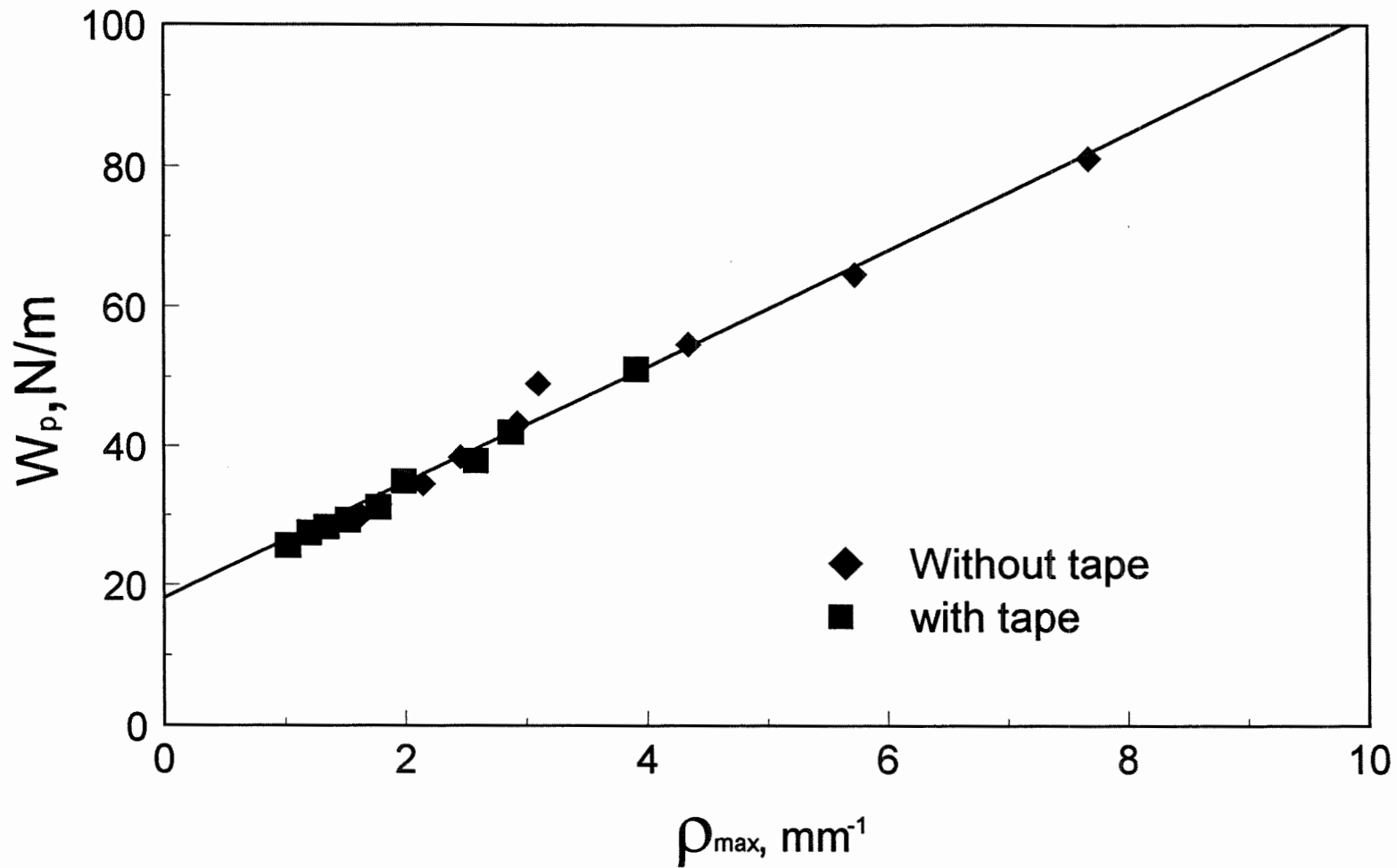


Figure 4.6. Variation of work of peeling, W_p , with r_{\max} for tope layer reinforced by adhesive tape (■) and without tape (◆).

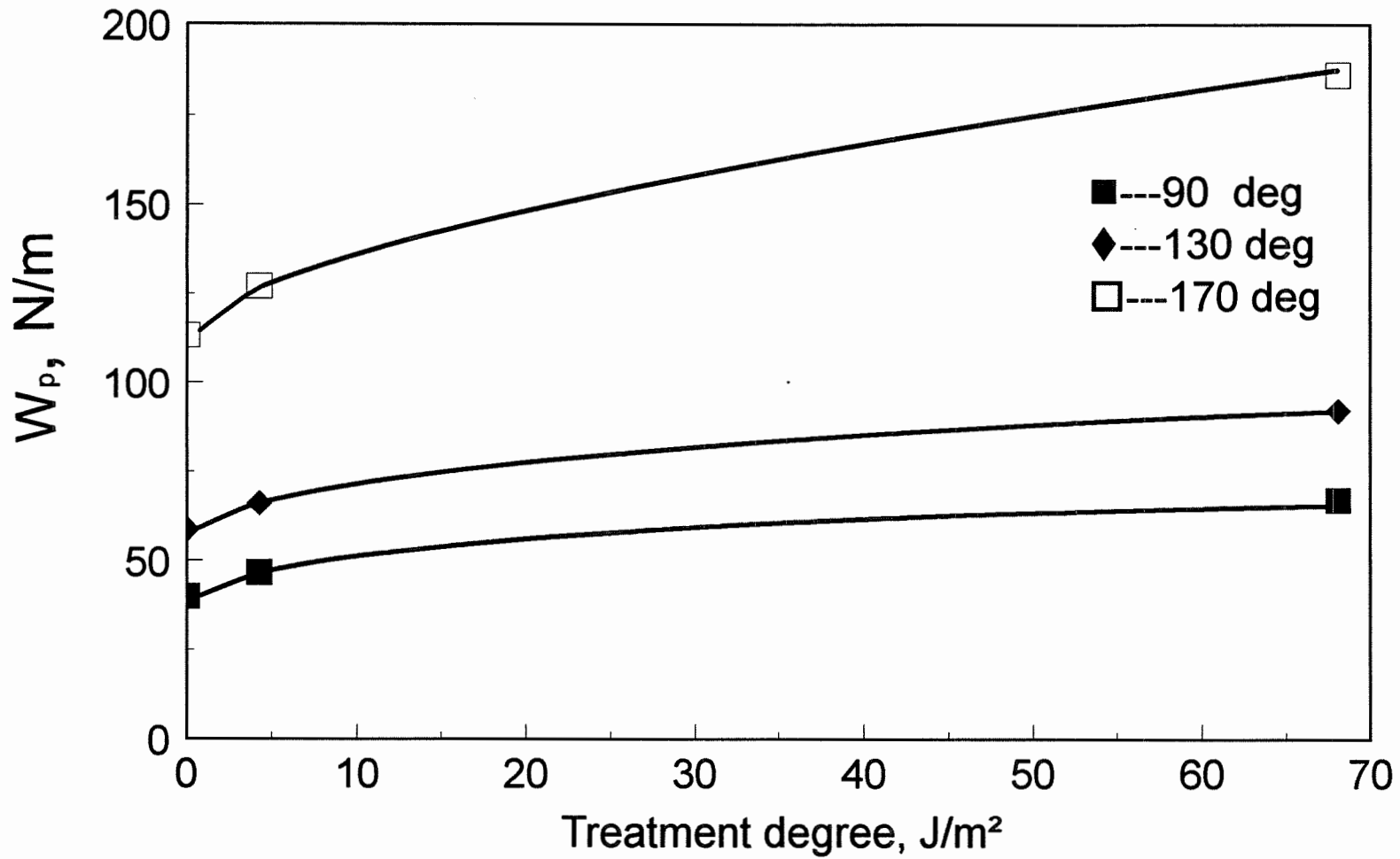


Figure 4.7. Variation of work of peeling, W_p , with the degree of corona treatment, at three different angles: 90° (■), 130° (◆) and 170° (□).

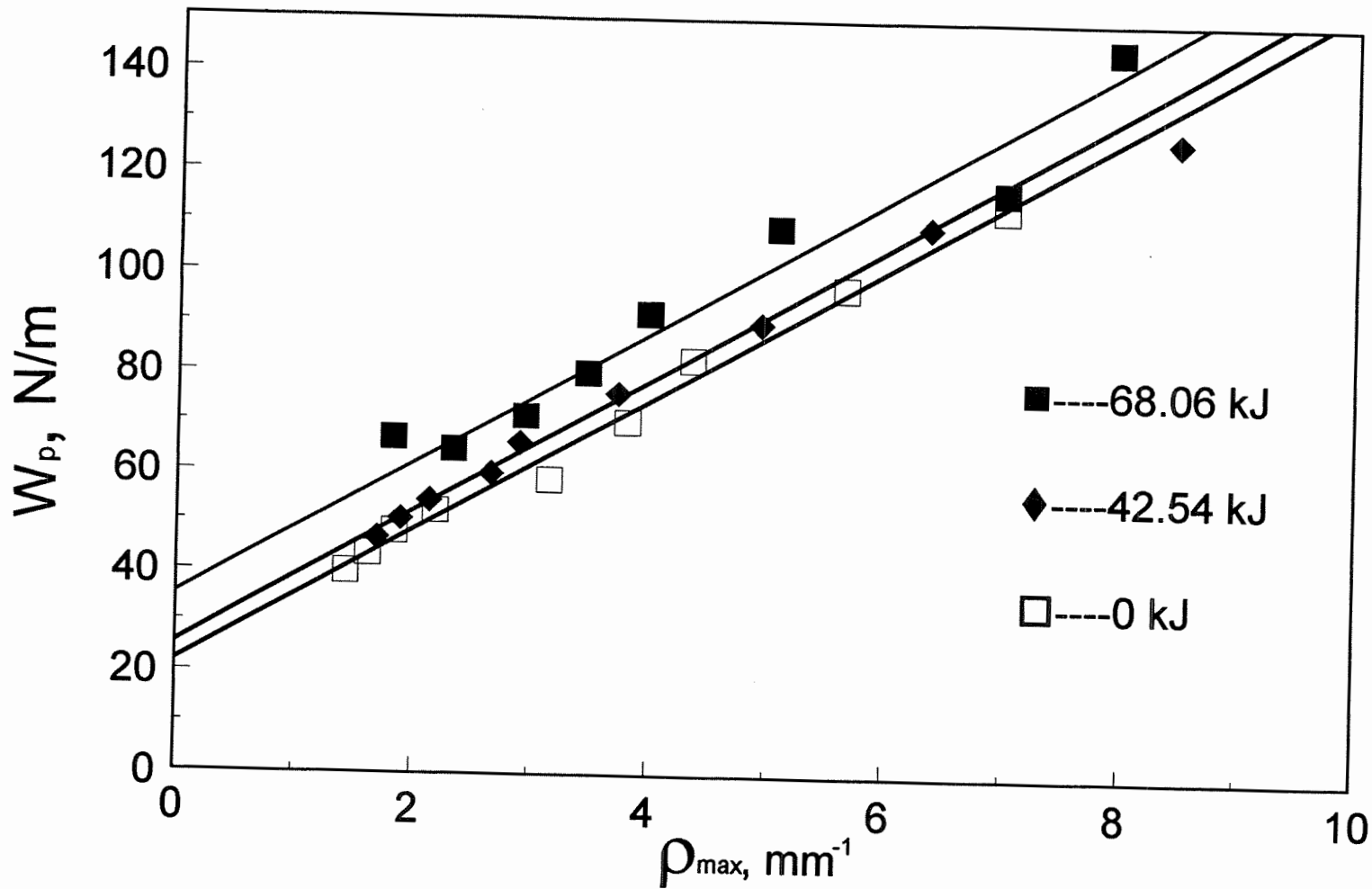


Figure 4.8. Variation of work of peeling, W_p , with ρ_{max} for three different corona treatments.

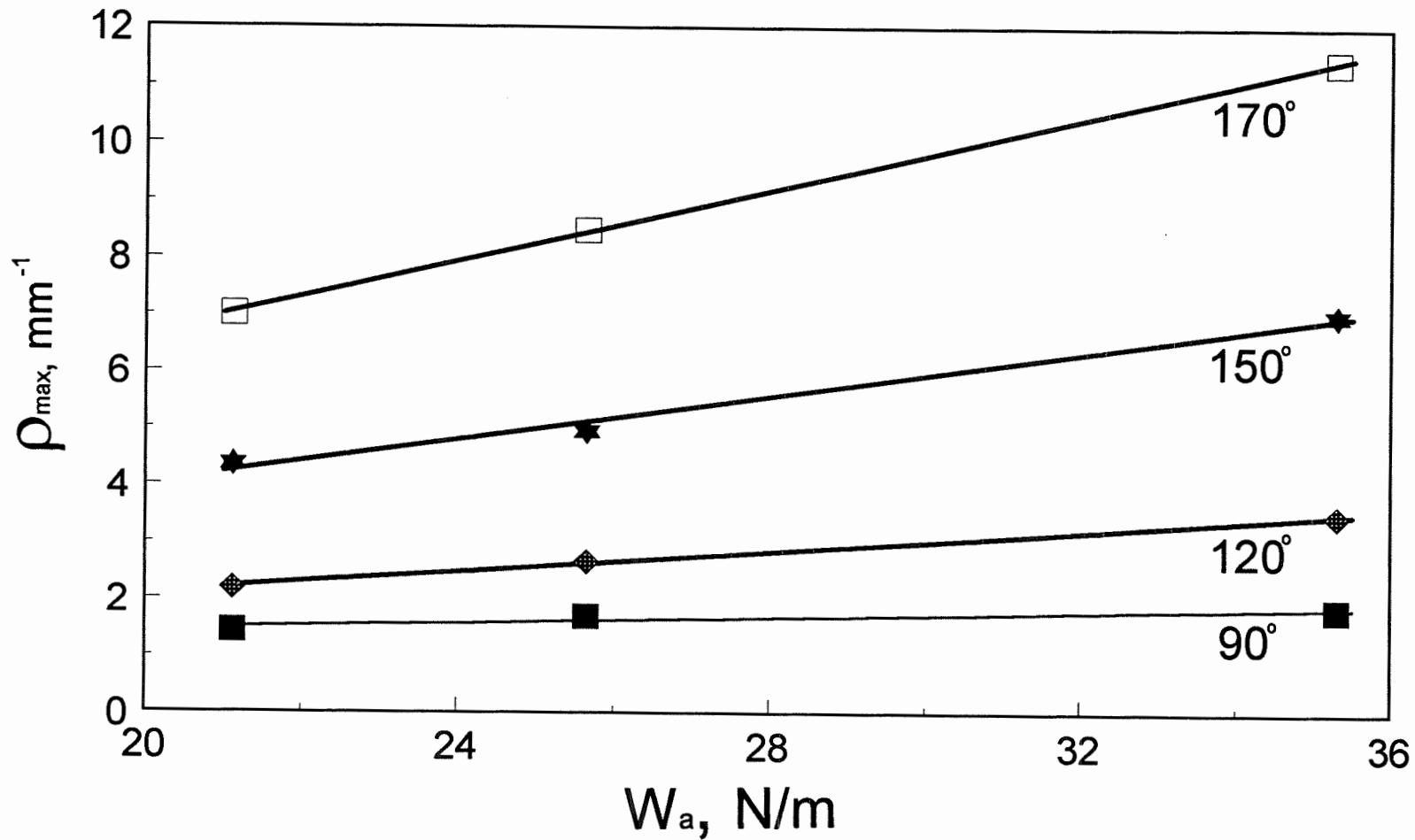


Figure 4.9. ρ_{\max} as a function of the work of detachment, W_a , at four different angles: 90° (■), 120° (◆), 150° (★), and 170° (□).

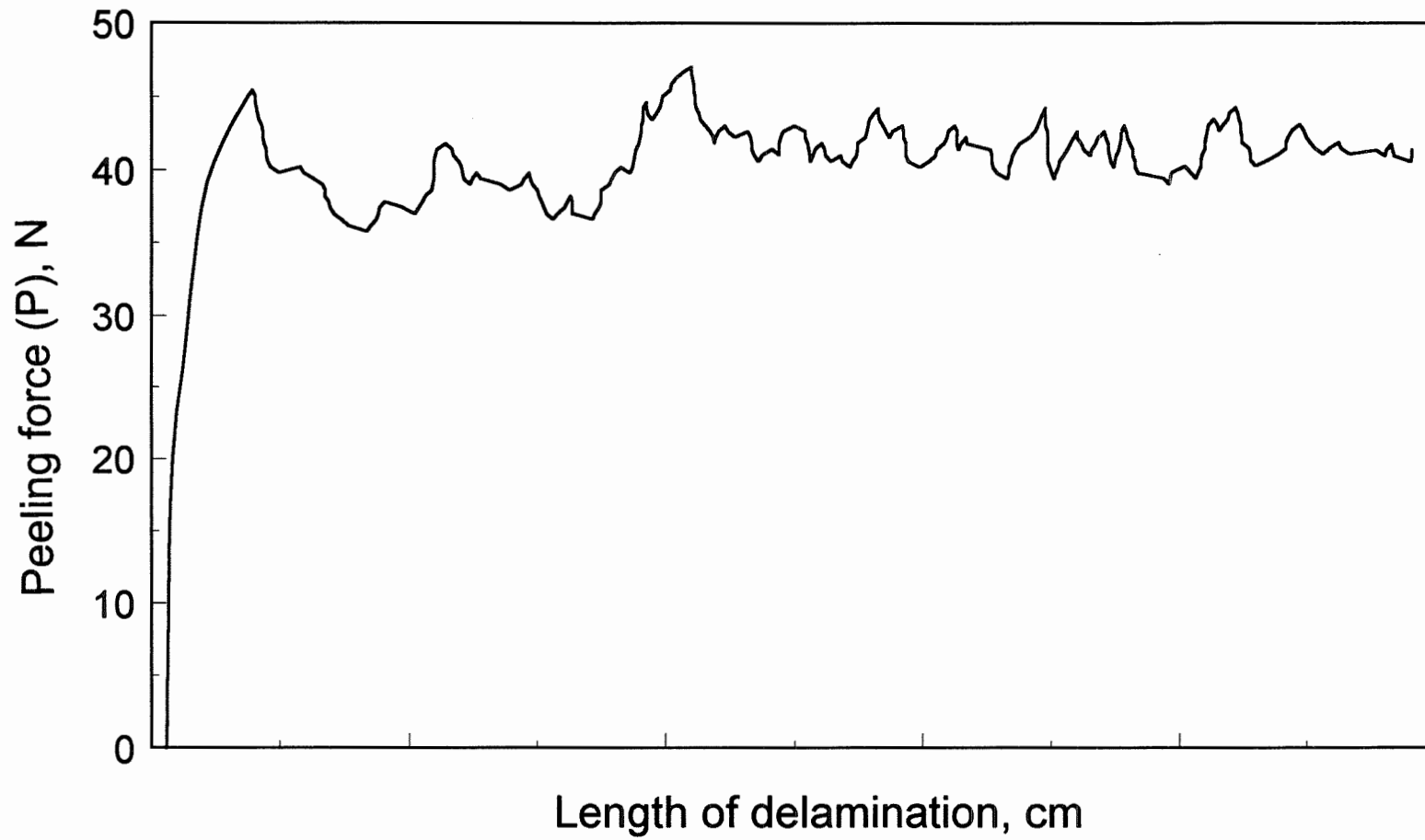


Figure 4.10. Force required for sample delamination at 90° and 2.54 cm/min.

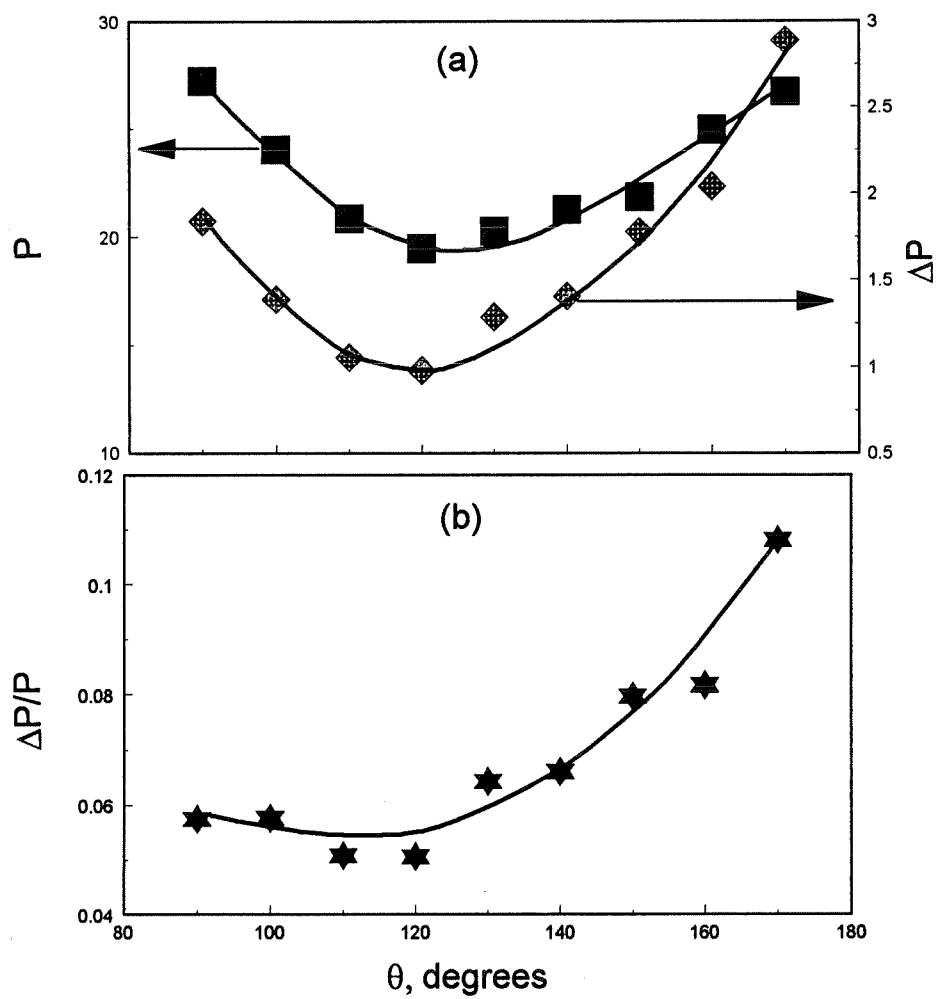


Figure 4.11. (a) Variation of peel force, P (■), and of absolute noise amplitude, ΔP (◆), with θ . (b) relative noise amplitude, $\Delta P/P$ (★), as a function of θ .

CHAPITRE 5

Angle-Dependent Delamination of Paper

Part IV: Effect of Beating on Bending Properties and Peel Test Results

Angle-Dependent Delamination of Paper
Part IV: Effect of Beating on Bending Properties and Peel Test Results

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ABSTRACT

We have investigated the effect of beating of fibres upon the work of peeling of paper and upon the contributions to this work: the work of detachment and the work of deformation. The work of detachment increases with beating level, but the work of peeling and the work of deformation show a maximum as a function of beating. The dependence of work of deformation on beating agrees well with predicted deformation work from the model based on in-plane tensile and compression data. The decrease of work of deformation and the work of peeling at high beating level may be attributed to the extensive modification of fibres, similar to that observed for in-plane properties.

5.1. INTRODUCTION

Beating is a common technique used in papermaking to enhance mechanical properties of paper. Its effect on the structural and physical properties of fibres, and, consequently, on paper properties has been the subject of much attention. It is a mechanical procedure which changes the morphology of cellulose fibres. The effects of pulp beating include fibre cracking, or opening of the cell wall, and fibre shortening. These lead to the formation of debris or fines and of external fibrillation [1 -7]. The cracking allows fibre swelling by breaking of some intrafibre hydrogen bonds and replacement by fibre-water hydrogen bonds [7] which cause internal fibrillation and further increases fibre flexibility. The term internal fibrillation refers to delamination of the fibre wall into separate lamellae. The swelling also produces straightening of kinks, crimps and curls at large and small scale which normally exist in the dried pulp fibres [5]. These changes increase sheet density by allowing fibres to conform more readily to each other, thus increasing bonded area. This results in the improvement both of out-of-plane and of in-plane mechanical properties of paper.

Out-of-plane mechanical properties are measured using tests such as the z-direction tensile test [8] and the Scott bond test [9]. Since most of the bonded areas of fibres are roughly parallel to the face of the sheet, the results of these tests characterize the bonding properties. Hence, the improvement of mechanical properties with beating has been attributed to the increase of bonded area. Recently, the effect of beating on out-

of-plane properties has been examined using the peel test. Skowronski and Bichard [10] reported that work of peeling, which they called "bond strength", increases with beating and wet pressing pressure, but z-bond toughness, which was called "specific bond strength" (SBS) and defined as the ratio of the total work to the area of bond broken, is independent of beating and of wet pressing pressure. In contrast, Retulainen and Ebeling [11], and Dasgupta [12] reported that SBS changes with beating.

We have recently shown [13,14] that work of peeling, W_p , results from two contributions: work of deformation, W_d , and work of detachment, W_a (the definitions of both W_a and W_d are given in Table 5.1). W_d and W_a may behave differently with beating, since the mechanisms involved are different. For laminated handsheets, W_a results from the breaking of fibre-fibre bonds at the interface [15], and reflects z-direction bond properties and their evolution with beating. W_d results from the propagation of the flexion, which is based on both in-plane-compression and in-plane-tension. It depends upon both fibre and bond properties and their evolution with beating as shown below.

In-plane mechanical properties are measured using tensile and compressive tests. For the tensile test, it is known that beating considerably improves the Young's modulus, tensile energy absorption (TEA) and tensile strength [5,12]. Several theories have been proposed to explain the effect of beating. They were based on the increase of the bonded area and the creation of fibrillation and of fines [16,17]. The most satisfactory explanation has been presented by Page [5,18]. This theory attributed at least half of the increase of the Young's modulus and tensile strength to the release of kinks, crimps and

curls. This permits the straight fibre segments to transmit the load. For the case of compression, studies of the effect of beating have focused on the compressive strength and on a description of the roles played by fibres and by bonds. Seth et al. [19] reported that the sheet compressive strength depends upon bonding strength at low degrees of bonding, but is controlled by fibre properties at high degree of bonding. Fellers [20] reported that at low degree bonding the compressive strength is governed by fibre buckling due to the free (unbonded) segment lengths in a sheet. At higher degrees of bonding, the free segment lengths are of the same order of magnitude as the fibre thickness. Compression failure is then controlled by the strength of the fibres.

The present study evaluates the effect of pulp beating on peeling of laminated handsheet in the light of the model proposed in ref [14]. The agreement between the experimental and theoretical results are also helpful to describe the effect of beating on flexural properties of paper.

Table 5.1. Nomenclature and definition of the principal terms used in this work.

term	Nomenclature	Definition	Units
M_b	bending moment	Moment per unit width to bend a strip from zero to a given curvature, ρ .	N
M_{ub}	unbending moment	Moment per unit width to unbend a strip from an initial curvature, ρ_0 to a given curvature ρ .	N
S_b	bending stiffness	Bending stiffness of the strip per unit width, defined as the initial slope of the curve M_b vs ρ .	N·m
S_{ub}	unbending stiffness	Unbending stiffness of the strip per unit width, defined as the initial slope of the curve M_{ub} vs $(\rho_0 - \rho)$.	N·m
M_{st}	moment strength	The highest value of moment during bending	N
W_b	work of bending	The work per unit area spent to bend the strip from zero to a given curvature, ρ .	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
W_{ub}	work of unbending	The work per unit area spent to unbend strip from a given curvature, ρ , to zero.	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
W_d	work of deformation	The work per unit area spent for the propagation of the flexion during peeling.	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
W_a	work of detachment	The work per unit area required to separate the top from the bottom layer.	$N \cdot m^{-1}$ or $J \cdot m^{-2}$

W_p	work of peeling	The total work per unit area measured during peeling.	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
	bond toughness	Work required to separate two bonded fibres per unit bonded area	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
TEA	tensile energy absorption	The ratio of the work spent during sample stretching, up to rupture, divided by sample surface.	$N \cdot m^{-1}$ or $J \cdot m^{-2}$
CSF	Canadian standard freeness	The rate at which a dilute suspension of pulp may be dewatered [21].	ml

5.2. EXPERIMENTAL

Handsheets were made from bleached kraft pulp refined to different levels in a PFI beater. In each case, water was added to 24g of dried pulp, so that the total mass was 240g. This 10% consistency suspension was placed in the beater. The beating level is defined as the number of revolutions of the beater. For this study, the beating levels used were 0, 500, $3 \cdot 10^3$, $6 \cdot 10^3$, $9 \cdot 10^3$ and $12 \cdot 10^3$ revolutions.

The handsheets were made in various ways, depending upon the test to be performed. For the peel and tensile tests, the handsheets were made by lamination of two handsheets, of $40 \text{ g} \cdot \text{m}^{-2}$ basis weight each, under wet pressure. The resulting handsheet

sample has basis weight of 40/40 g·m⁻². For in-plane tensile and compression test, single handsheets of 400 g·m⁻² basis weight were used.

The delamination was performed with a peel test apparatus (IMASS Model SP-103B). In-plane tension and compression measurements were performed using an Instron tester. All of these measurements are discussed elsewhere [14].

5.3. RESULTS AND DISCUSSION

The effect of beating upon apparent sheet density, Young's modulus, tensile and compressive strength, and Canadian standard freeness (CSF) are presented in Table 5.2. All measured properties of paper were affected by the level of beating. Their variations were consistent with previously published work [5,11,12,18].

Table 5.2. Physical properties of dried bleached kraft pulp used in this study.

Beating level, *10 ³ rev in PFI	Unbeaten	0.5	3	6	9	12
C. S. Freeness, ml	656	609	510	407	310	215
Apparent density, g·cm ⁻³ .	0,52	0,60	0,66	0,72	0,75	0,85
Young's Modulus, E, MPa.	2076	3446	3873	4271	4283	4443
Tensile strength, N·m·g ⁻¹ .	35,55	69,10	77,53	78,92	81,11	71,25
Compressive strength, N·m·g ⁻¹ .	7,87	15,02	18,70	24,58	23,82	17,00

5.3.1. Peel test results

Fig. 5.1 shows the variation of work of peeling, W_p , with beating level for three different peeling angles. In all three cases, W_p increases to a maximum (at $9 \cdot 10^3$ rev in PFI mill) and then decreases for higher beating levels. The decrease of W_p at high beating level was observed only for laminated handsheets and depended on peeling angle. In contrast, for delamination of single handsheets, W_p increases with beating, and then levels off at the maximum [10].

The variation of W_p with beating, for laminated handsheets, may be understood from its contributions, W_a and W_d . To achieve this goal, Fig. 5.2 shows the variation of W_p as a function of the measured curvature, ρ_{max} , for the different beating levels. Each set of data, of a given beating level, presents a straight line which can be described by $W_p = \alpha_p \cdot \rho_{max} + W_a$ [14]. The slope, α_p , characterizes the dependence of W_d upon ρ_{max} , and the intercept, W_a , is the work of detachment. The values of α_p and W_a determined from the curves are presented in Table 5.3. The observed increase of W_a with beating level may be attributed to the increased bonded area [22]. In contrast, α_p presents a broad maximum between $3 \cdot 10^3$ and $6 \cdot 10^3$ rev in the PFI mill. In what follows we shall investigate the origin of this maximum.

The work of deformation, W_d , proportional to α_p , is dissipated during the propagation of flexion. This involves only the in-plane tensile and compressive properties of paper [14]. Knowledge of the effect of beating upon these properties, and upon the bending and unbending moments are required in order to explain the effect of beating upon W_d . The variation of Young's modulus, and of tensile and compression strength are given in Table 5.2. We see that Young's modulus increases continuously with beating, but that tensile and compressive strength present maxima in the region in which α_p is maximum. Similar dependencies of sheet strength were observed by Retulainen and Ebeling [11], and Dasgupta [12]. They attributed the decrease of the above parameters at high beating level to degradation of fibres, which in turn reduce the sheet strength.

Table 5.3. Effect of beating upon work of detachment, W_a , and work of deformation represent here by the slope α_p .

Beating level, $\cdot 10^3$ rev in PFI	Unbeaten	0.5	3	6	9	12
$\alpha_p, \cdot 10^{-3}$ N	11,24	13,90	14,70	14,96	12,98	7,97
$W_a, \text{N}\cdot\text{m}^{-1}$	11,95	21,21	28,64	30,27	38,87	42,19

5.3.2. Effect of beating upon bending and unbending moment

Bending (M_b) and unbending (M_{ub}) moments result from distribution of the stress throughout the thickness. They may be calculated from in-plane stress-strain data in tension and in compression following the procedure and equations developed elsewhere [14]. Thus, M_b and M_{ub} are the link between in-plane properties and the work of deformation. They may also reveal the effect of beating upon flexural properties of paper.

Fig. 5.3 shows the variation of M_b as a function of the curvature, ρ , for different beating levels. Each curve is composed of elastic and inelastic regions. The elastic region is characterized by a linear variation of M_b with ρ , where the slope, S_b , represents the stiffness. The inelastic region is characterized by a pronounced maximum (bending strength, M_{st}) at the first stage of irreversible flexion, followed by a practically constant value of M_b . The effect of beating is different depending upon whether M_b is in the elastic or inelastic regions. In the elastic region, M_b decreases with beating, as may be seen from the variation of S_b reported in Table 5.4. In the inelastic region, M_b has maximum for $6 \cdot 10^3$ rev. (in PFI mill), the beating level for which we also observe the maximum for in-plane strength and for α_p . The effect of beating in the inelastic region can be represented by the moment strength, M_{st} as shown in Table 5.4.

Table 5.4. Variation of bending stiffness, moment strength and the curvature at the moment strength as a function of beating level.

Beating level, $\cdot 10^3$ rev in PFI	Unbeaten	0.5	3	6	9	12
Bending stiffness, S_b , N·mm.	0,11	0,10	0,09	0,08	0,07	0,05
Bending stiffness, $S_b = E \cdot t^3 / 12$, N·mm.	0,09	0,10	0,08	0,07	0,05	0,04
Moment strength, M_{st} , $\cdot 10^3$ N	5,85	11,17	10,9	13,95	12,45	9,57
Curvature at the moment strength, mm^{-1} .	0,06	0,17	0,20	0,32	0,31	0,31

We have also measured the thickness of the sheets, which decreases with beating. Using the values of E from Table 5.2, we may calculate S_b using the equation $S_b = E t^3 / 12$ [23, 24]. These are in reasonable agreement with experimental values, as shown in Table 5.4. Hence, we may attribute the decrease of S_b to the decrease of the thickness with beating which out-weights the increase in E .

The variation with beating of M_b in the inelastic regime (represented by M_{st} , in Table 5.4) may be separated into two parts. First, the increase to maximum may be related to enhancement of mechanical properties of paper in both the tensile and compressive modes. This has been attributed to the increase of bonded area and the straightening of fibres [5, 18]. The subsequent decrease, beyond the maximum, may result from damage to the fibre structure and to the fibre cell walls [1, 25] which leads, in turn, to a reduction of both tensile and compressive strength of fibres.

Fig. 5.4 shows the variation of unbending moment, M_{ub} , with curvature, $\rho' = (\rho - \rho_0)$, for different beating levels. The effect of beating upon M_{ub} is similar to that observed for M_b . By analogy to bending, we can define the unbending stiffness S_{ub} and moment strength M'_{st} . The dependence of S_{ub} and M'_{st} on beating is similar to that observed for bending, as it can be seen from Figs 5.3 and 5.4.

5.3.3. Effect of beating upon work of deformation, W_d

The work of deformation, W_d , represents the energy expended for propagation of the flexion in the peel test. We have shown [14] that W_d is composed of the work of bending, W_b , and that of unbending, W_{ub} .

W_b (or W_{ub}) may be calculated using the data for the bending moment, $M_b(\rho)$ (or of unbending moment, $M_{ub}(\rho')$) following the procedure developed in ref. [14]. Both W_b and W_{ub} are zero for small values of ρ_{max} (elastic region) and approximately linear functions of ρ_{max} for the rest (inelastic region). They can be written as [14]:

$$W_b = \alpha_b \cdot \rho_{max} + \beta_b \quad \text{and} \quad W_{ub} = \alpha_{ub} \cdot \rho_{max} + \beta_{ub}.$$

The deformation work, the sum of W_b and W_{ub} , will be an approximately linear function of ρ_{max} and can also be written as $W_d = (\alpha_b + \alpha_{ub}) \cdot \rho_{max} + (\beta_b + \beta_{ub}) = \alpha_d \cdot \rho_{max} + \beta_d$. The values of the different slopes and intercepts and their variation with beating are presented in Table 5.5.

Table 5.5. Effect of beating on calculated values of the slopes and intercepts of W_b , W_{ub} and W_d in the inelastic range, expressed as a function of ρ_{max} , for a strips of $40 \text{ g}\cdot\text{m}^{-2}$ basis weight, and the experimental slope, α_p , determined from peel test results.

Beating level $\cdot 10^3 \text{ rev.}$	Slopes, $\text{N}\cdot 10^{-3}$.				Intercepts, $\text{N}\cdot\text{m}^{-1}$.		
	α_b	α_{ub}	$\alpha_d = \alpha_b + \alpha_{ub}$, calculated	α_p , from peel test	β_b	β_{ub}	$\beta_d = \beta_b + \beta_{ub}$
0	4,57	1,85	6,42	11,24	-0,14	0,22	0,09
0,5	8,61	3,49	12,10	13,90	-0,97	-3,36	-4,34
3	8,50	3,32	12,13	14,70	0,45	1,01	1,46
6	8,49	3,80	12,29	14,96	0,17	-0,59	-0,42
9	7,29	2,58	9,87	12,98	-0,25	0,13	-0,13
12	5,70	2,64	8,34	7,97	-0,3	-0,13	-0,46

The presented slopes α_b , α_{ub} and α_d in Table 5.5 show an increase to a broad maximum. At high beating level ($9\cdot 10^3$ rev. and above) they decrease. The calculated intercepts show no systematic variation.

These observations may be understood on the basis of the same model proposed to explain the variation of tensile and compressive properties, and to explain the moment strength. Beating improves mechanical properties of paper by increasing bonded area and straightening fibres. However, at high degrees of beating fibres are degraded reducing the fibre strength and their contribution to the work of deformation.

The values of the calculated slopes, α_d , are in reasonable agreement with the experimental values, α_p , except for unbeaten pulp where α_p is almost twice α_d . The agreement between α_p and α_d for beating levels between $0,5 \cdot 10^3$ rev and $12 \cdot 10^3$ rev leads us to conclude that our model and explanation are correct. The dependence of works of peeling, detachment, and deformation on beating level is presented in Fig. 5.5. As it is seen, the work of detachment, W_a , increases with beating. The decrease of the work of peeling at high beating level observed in Fig. 5.1 and 5.5 may be attributed to the fact that this increase is not enough to balance the decrease, due to deterioration of fibres, of the work of deformation, W_d especially at large angles.

The discrepancy between α_p and α_d for unbeaten pulp may be attributed to the in-plane properties of unbeaten pulp, that is, to stretching. Page and Tydeman [5,18] reported that sheets made from dried and unbeaten pulp contain fibres with kinks, crimps and curls which are strengthened progressively during stretching. Thus, the plastic stretch contribution of strain, ϵ'_p , due to the total strain, ϵ' , will be important. Skowronski and Robertson [26] have reported measurement which are in agreement with Page's theoretical prediction. The effect of this in-plane property on the discrepancy between α_p and α_d may be attributed to bending strain as follows.

Our model [14] for bending strain, ϵ , was developed taking into account the plastic deformation at the neutral surface. The value of ϵ was approximated as:

$$\varepsilon = \frac{z}{R}(\varepsilon_p + 1) + \varepsilon_p \approx \frac{z}{R}$$

where R is the radius of flexion at the neutral surface, z is the distance through the thickness from this surface, and ε_p is the plastic strain at the neutral surface. In [14] it was found that ε_p was negligible. This is not correct for unbeaten pulp. Thus a more complicated expression would be necessary to describe paper made of unbeaten pulp.

5.4. CONCLUSION

The work of peeling of laminated handsheets shows a maximum as a function of beating. The contributions to this work, the work of deformation and the work of detachment, behave differently with beating. The work of detachment increase with beating due to the increase of the bonded area between fibres. The work of deformation shows a maximum as a function of beating. The calculated work of deformation from in-plane tensile and compression data shows that the decrease of W_d above the maximum is due to the damage to fibre walls, which reduces its in-plane mechanical properties. Hence W_d and W_p are due not only to bond breaking, since they result partially from the deformation of fibres in tension and in compression. This conclusion may explain the decrease of the specific bond strength with beating observed by Retulainen and Ebeling [11] and Dasgupta [12]. The effect of the fibre contribution on the work of peeling may also be revealed by the effect of wet pressing pressure on the peel test results.

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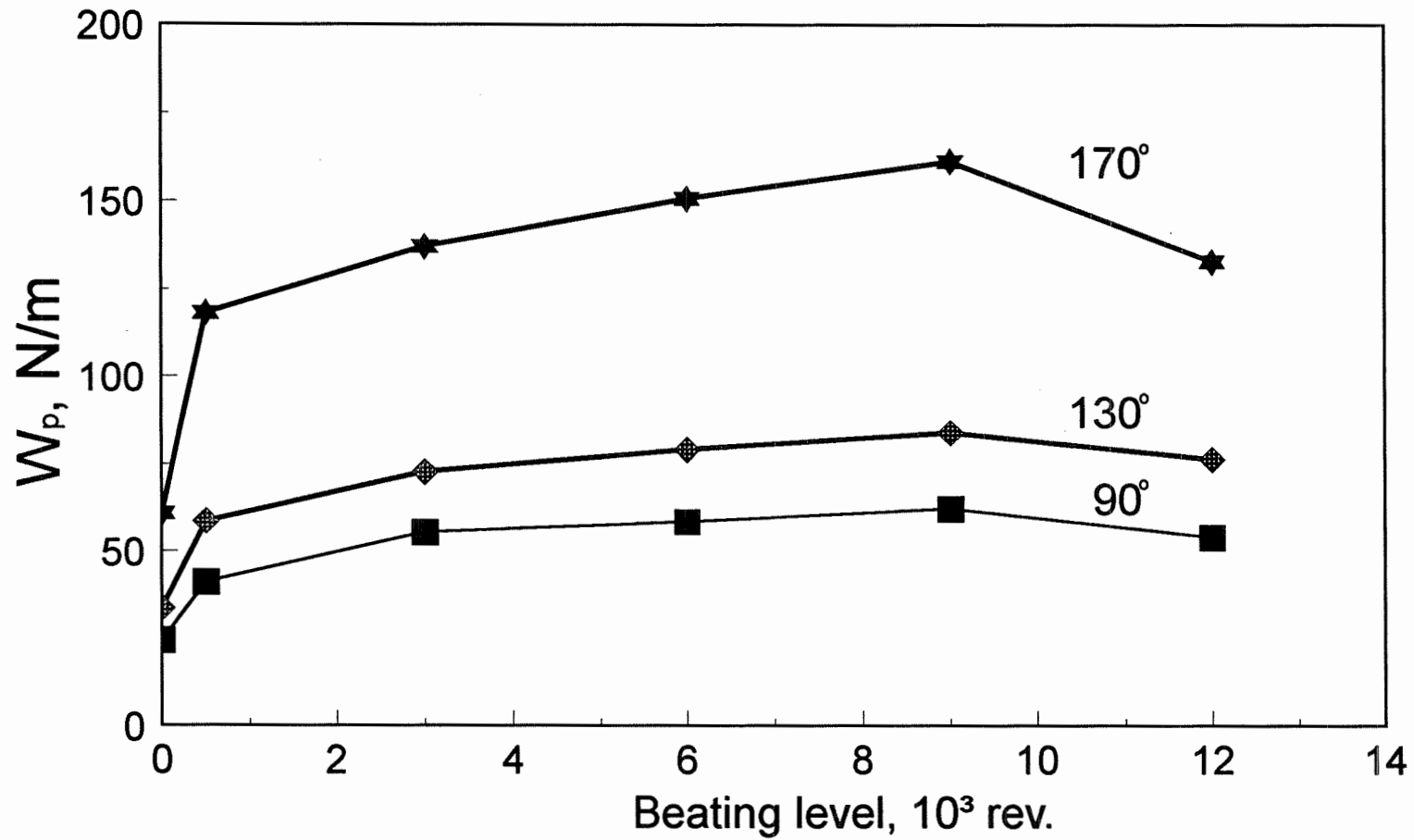


Fig. 5.1: Variation of the peel work, for laminated handsheet of 40/40 g/m^2 basis weight, with beating at three different angle (90°, 130° and 170°).

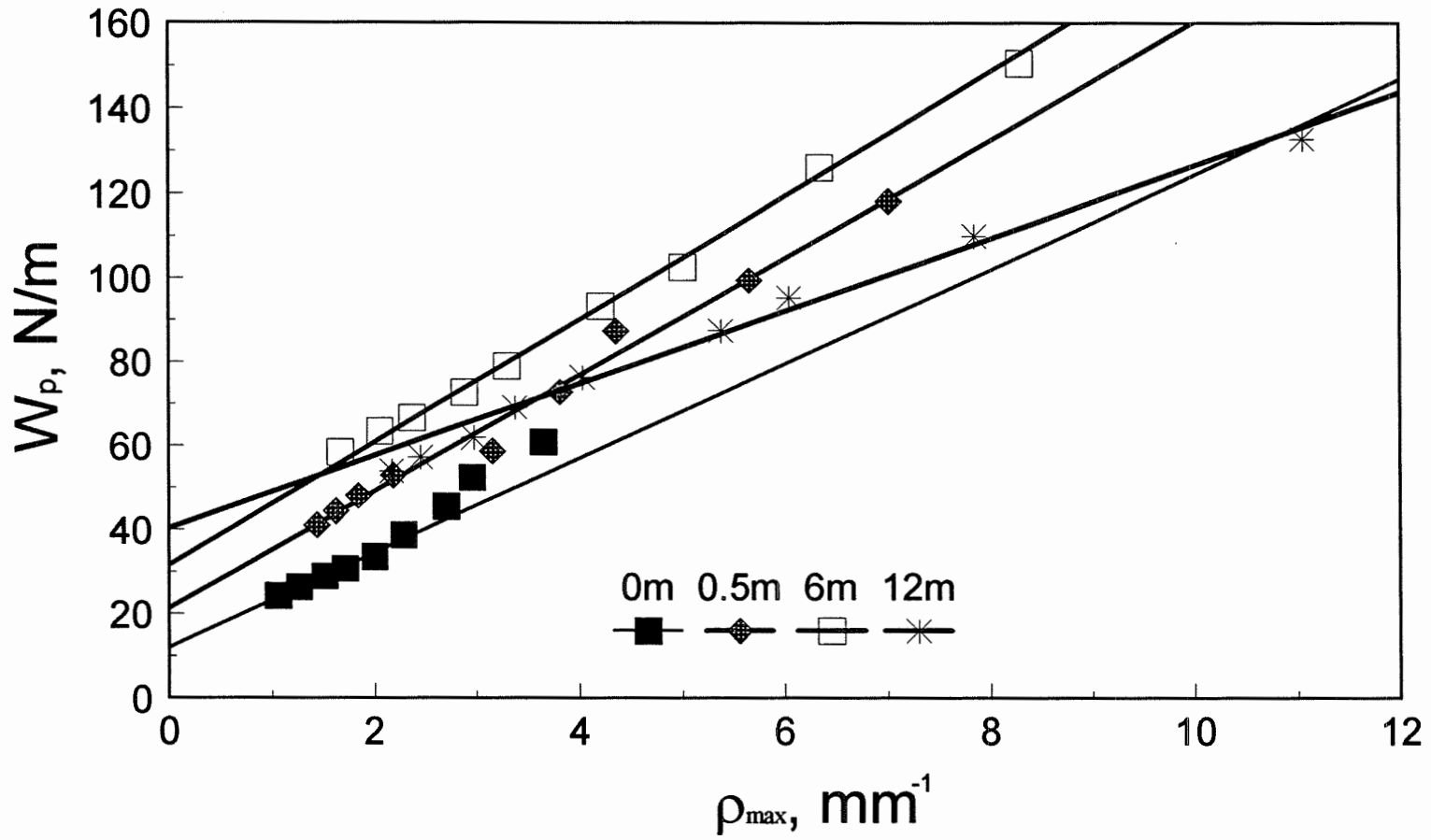


Fig. 5.2: Variation of peel work, W_p , with curvature, ρ_{max} , for different beating level.

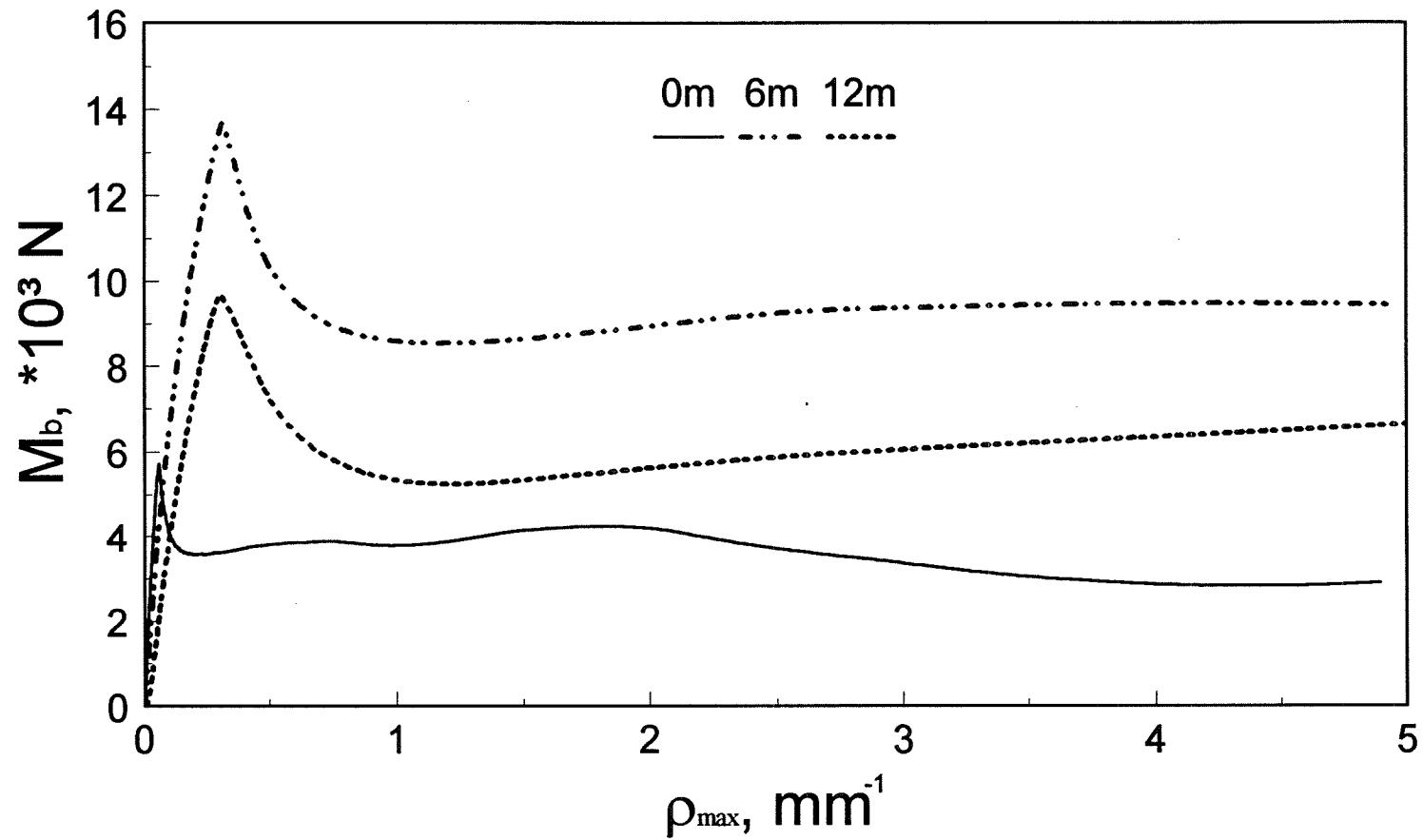


Fig. 5.3: Variation of calculated bending moment with curvature for three different beating level (0, 6×10^3 and 12×10^3 rev. in PFI mill). The thickness used for the calculation is that measured for handsheet of 40 g/m^2 basis weight.

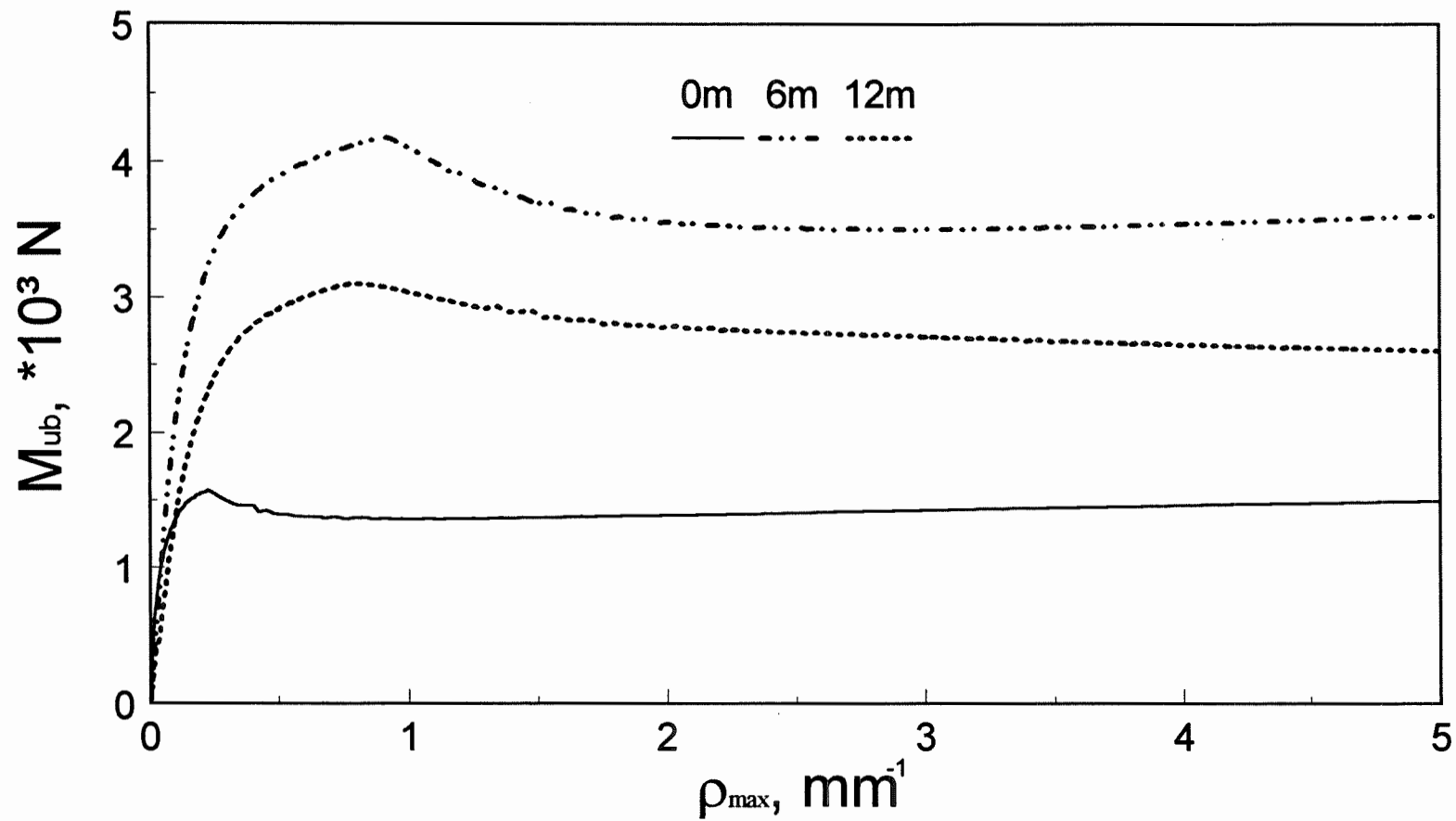


Fig. 5.4: Variation of calculated unbending moment with initial curvature for three different beating level (0, 6×10^3 and 12×10^3 rev. in PFI mill). The thickness used for the calculation is that measured for handsheet of 40 g/m^2 basis weight.

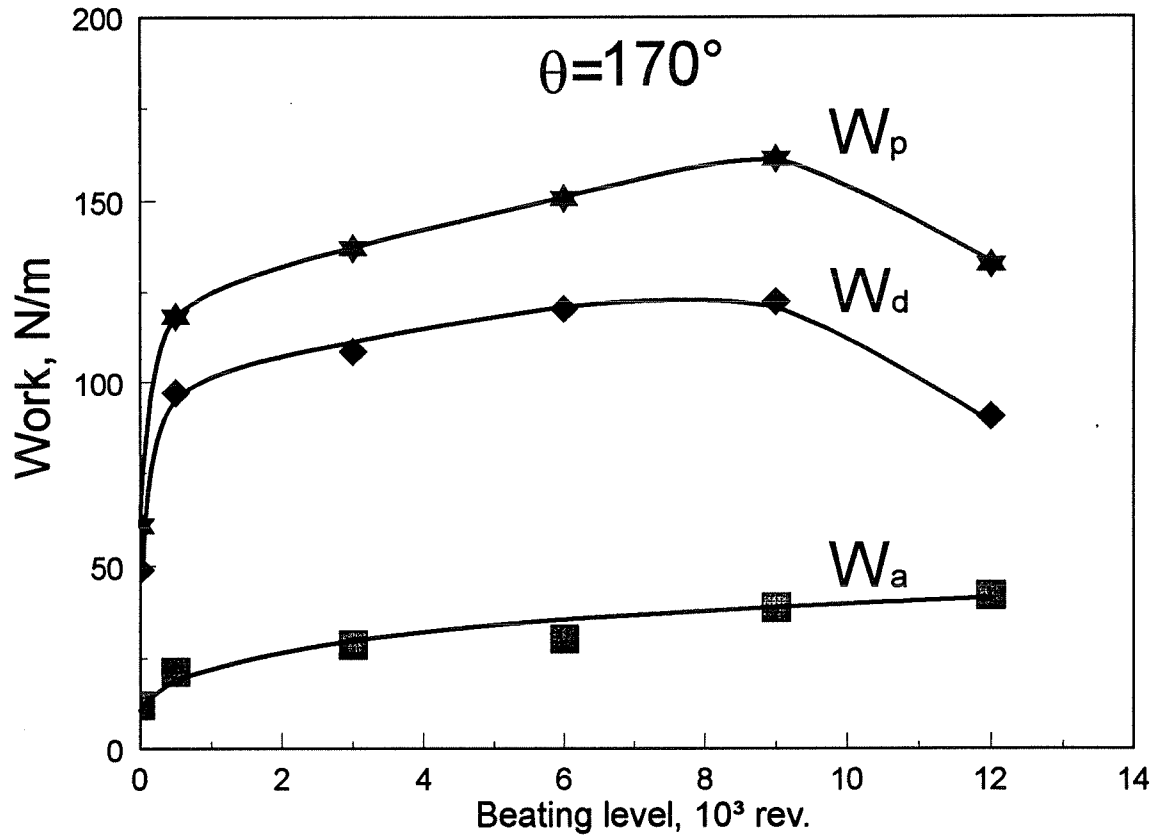


Fig. 5.5: The works of peeling, detachment, and deformation as a function of beating level. The peeling angle is $\theta=170^\circ$.

CONCLUSION

Le papier se délamine de façon inélastique, c'est-à-dire que la partie qui se détache se déforme de façon irréversible durant la flexion. Il en résulte que l'énergie de pelage, W_p , est la somme de l'énergie de détachement, W_a , et de l'énergie de déformation, W_d .

L'énergie de déformation, W_d , est dissipée durant la propagation de la flexion. Elle augmente avec l'épaisseur de couche supérieure (couche à délaminer), l'angle et la vitesse de pelage. Un modèle théorique basé sur l'analyse de la propagation de la flexion, utilisant les courbes expérimentales contrainte-déformation en traction et en compression dans le plan, nous a permis de calculer l'énergie de déformation. Les résultats ont montré que W_d est pratiquement une fonction linéaire de la courbure de la flexion. W_a

L'énergie de détachement, W_a , est dissipée dans la zone de séparation, et est indépendante de la courbure (donc de l'angle). Elle est aussi indépendante de l'épaisseur des couches laminées (ou de leurs densités massiques de surface) et de la variation de la courbure (due à la variation de la rigidité) de la couche supérieure. Cependant, elle varie avec les traitements qui affectent la liaison interfibre, tels que le raffinage et le traitement par effet couronne appliqués à l'interface des échantillons laminés. W_a est donc caractéristique de l'interface, et spécialement des liaisons interfibres à l'interface. Étant donné que pour les angles $90^\circ \leq \theta \leq 180^\circ$ la rupture dans la zone de séparation suit le mode

I (clivage), W_a peut être attribuée à la rupture des liaisons interfibres dans la direction normale au plan de la liaison.

L'énergie de déformation fait intervenir les propriétés de traction et de compression dans le plan. Il en résulte que W_d est due aux contributions des fibres et des liaisons interfibres. La contribution des liaisons interfibre provient essentiellement de leurs ruptures qui se produisent dans le mode II (en cisaillement) durant la traction, et dans un mélange de mode I et de mode II durant la compression. Par conséquent, l'énergie de pelage, $W_p = W_d + W_a$, ne peut pas être attribuée exclusivement à la cassure des liaisons interfibre et plus spécialement à la cassure des liaisons dans le mode I, tel que fut le cas auparavant. Cependant, il est possible de trouver des conditions où l'énergie de déformation sera négligeable devant l'énergie de détachement et que la rupture dans la zone de détachement se fera suivant le mode I (cassure des liaisons dans la direction normale au plan). Par exemple, (i) appliquer un ruban adhésif à la couche supérieure qui augmente sa rigidité élastique tout en ne participant que de façon élastique au processus de pelage, (ii) utiliser une couche supérieure moins épaisse, pour réduire W_d , tout en étant assez épaisse pour prévenir la pénétration de l'adhésif jusqu'à l'interface. L'augmentation de la rigidité de la couche supérieure est sans effet sur W_a .

Dans les conditions des paragraphes précédents (W_d négligeable devant W_a et W_a résultant de la cassure des liaisons interfibres à l'interface des échantillons laminés), l'énergie de pelage peut être approximée à W_a et utilisée pour la détermination de la ténacité de la liaison interfibre (travail par unité de surface de la liaison). La méthode est

d'une grande utilité, car elle permettra la détermination de la ténacité de la liaison interfibre dans la direction z "z-bond toughness" dans le papier. Les résultats ne seront pas affectés par la concentration de la contrainte et l'effet de l'adhésif, comme c'est le cas pour la traction dans la direction z.

L'amélioration de la liaison interfibre par traitement couronne nous a permis de montrer que W_d est fonction de l'énergie de détachement W_a , et ainsi établir l'effet amplificateur de W_d durant le processus de pelage. Le phénomène d'amplification observé peut expliquer, dans un premier temps, la sensibilité de la méthode vis-à-vis la variation de W_a , et dans un deuxième temps, l'origine et l'importance des fluctuations observées durant la mesure de la force de pelage.

Le pelage s'est avéré sensible aux propriétés rhéologiques du papier. En effet, la rigidité à la flexion et l'énergie de déformation varient avec le raffinage. Nous prévoyons que la méthode sera utile pour l'étude de l'effet de la pression humide sur les propriétés de flexion du papier.

Nous envisageons donner suite à ce travail en étudiant l'effet de l'épaisseur de la couche supérieure sur W_d et l'effet de la vitesse sur les deux types d'énergies (W_a et W_d) dont une publication est en cours de préparation. Les résultats de ces études futures nous seront utiles pour développer un modèle qui permettra de faire la séparation entre les énergies W_a et W_d , connaissant simplement la variation de la force avec l'angle de pelage.

Nous prévoyons aussi généraliser le modèle pour décrire le pelage des échantillons non laminés. Ceci sera possible en tenant compte du fait que le système tend à minimiser

son énergie durant le délaminage en se fracturant suivant le plan qui offre la résistance minimale à la rupture, tout en minimisant l'énergie de déformation par une réduction de l'épaisseur détachée. Cette étude pourrait être utile pour étudier la variation de la densité de la liaison interfibre dans l'épaisseur.

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