UNIFIED PAPER SHEET, PROCESS OF MAKING, AND PRESSURE-SENSITIVE ADHESIVE TAPE MADE THEREFROM

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Inventor: Isaac R. Dunlap

Attorney: Charles A. Harris

INVENTOR
ISAAC R. DUNLAP

PARER SHEET, PRESSURE-SENSITIVE, ADHESIVE TAPE MADE THEREFROM

Drawing 1

Drawing 2

Drawing 3

Adhesive

Backing

INVENTOR
ISAAC R. DUNLAP

ATTORNEY
Charles A. Harris
This invention relates to improved unified papers suitable for many uses, particularly to unified papers useful as backings for adhesive tapes, sandpapers and the like, and in artificial leathers, and to improved pressure-sensitive adhesive tapes and other products made with the improved backings; as well as to improved methods for manufacturing such papers.

This application is a continuation-in-part of my co-pending applications Serial No. 624,144 filed November 23, 1956, and Serial No. 89,687 filed February 16, 1961, both of which have since become abandoned.

It is most important that the fibers in papers of this type be unified or held together so that the paper possesses satisfactory resistance to delamination and fiber picking and will not blister or crack when flexed. For instance, paper backed pressure-sensitive adhesive tapes commonly are made by coating one side of the backing with an adhesive and then winding the tape upon itself with the coated side facing inward to form a roll. If a liner is not used, or some other special precaution is not taken, when the tape is rapidly unwind, the backing will be split into plies or delaminated because the adherence of the outside of the backing to the adhesive on the inside of the tape being unwind is greater than the coherence of the fibers in the backing.

The most common method of "unifying" the paper or strengthening it against splitting, is to incorporate a "unifying agent," commonly termed an "impregnant," in the web to bond the fibers together. Various extensible polymeric materials such as rubbery polymers and mixtures thereof have been used for this purpose. Such impregnants allow the sheet to retain a major portion of the characteristic flexibility of a paper web and yet bind the fibers against delamination of the backing when a roll of pressure-sensitive tape is unwound, for example. Normally sufficient unification to prevent delamination can be attained if it is possible to incorporate sufficient impregnant in the web. Typically, the web is saturated with the impregnant to provide the necessary unification. Since the cost of the impregnant is relatively high, one disadvantage of this approach is apparent. With some tapes and certain other products, such as artificial leathers, in which high delamination resistance is desirable, it may not be possible or even desirable to incorporate into the web the necessary amount of impregnant to prevent delamination. For these and other reasons, this approach to solving the problem of providing the necessary delamination resistance leaves much to be desired.

In addition to the delamination resistance, or internal strength, necessary in a paper backing for pressure-sensitive adhesive tape, for example, it is necessary that the backing also possesses the necessary strength, flexibility and elongation to enable curing the tape without tearing when applying it to a surface in a desired configuration, and sufficient flatness to lie against the surface to which it is applied. Improvements in tear strength of such paper-backed pressure-sensitive tapes can be obtained, though to a relatively small degree, by the use of soft and more highly extensible polymeric materials in the impregnant. The use of soft polymers in flat papers also increases to some small degree the elongation which can be obtained in the finished tape. However, the overall conformability of such a flat sheet is inadequate for many applications and results in tearing of the backing. Exemplary is the application of paper-backed pressure-sensitive tapes in masking a painted surface to which will be applied to a curved painted design.

Hence, to obtain a tape having the necessary delamination resistance, tear strength, flexibility and elongation, it has been common to rely upon the mechanical technique of creping the paper backing prior to impregnation. Creping may be described as a stepwise disruption of the starting web by the intermittent application of force to the web while the web is free to move at right angles to the plane of the web. This results in an opening up of the web and the formation of relatively large undulations or crinkles in the web, itself. The web is "opened up" by movement of the overlying fibers of the web away from one another in spaced areas of the web to increase the size and perhaps the number of the interspaces between them in these areas. It is this openness that provides sufficient saturaibility, or allows the incorportion of enough impregnant, to provide the necessary delamination resistance. Again, this requires a high percentage of relatively costly impregnant and consequently is disadvantageous from a cost point of view.

Moreover, while the increased by the mechanical technique of creping and normally enough improvement in saturaibility may be obtained to provide the necessary delamination resistance, a creped masking tape backing, for example, has some marked disadvantages such as high overall caliper, tendency of the paint to bleed under the tape at the points of undulations and to build up at the edge of the tape, limited ability to form curved lines and conform to curved surfaces, and the like.

The prior art teaches that to obtain maximum delamination resistance in conventional saturating papers, the starting web must be relatively open with the fibers spaced apart in order to be able to receive a relatively large amount, or high level, of impregnant. In fact, all authorities in the art up to as late as four years after the filing date of application Serial Number 624,144, now abandoned are agreed that for proper unification, the raw paper used in preparing unified backings should be selected from highly porous, highly absorptive bimolecular papers of low bulk density approaching blotting paper in texture; and that in forming such papers or in manipulating them prior to or during unification, any operation that substantially alters the foregoing characteristics would detrimentally affect the results obtained in unification.

I have discovered that by going in the opposite direction, i.e., compacting the starting web in a particular way and impregnating it, I can obtain a unified paper sheet possessing unexpectedly improved properties. I have discovered that by compacting the web horizontally with respect to its faces while applying force acting perpendicularly to said faces to resist substantial wrinkling of the web body, and impregnating or incorporating a unifying agent in the web; I can obtain a unified paper sheet possessing the following properties:
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1. Unexpectedly high delamination resistance at low cost or unexpectedly great cost savings at prior art levels of delamination resistance;
2. Extraordinary conformability, i.e., ability to be conformed smoothly and easily to curved surfaces and other three dimensional objects and to be smoothly applied in different shapes to flat surfaces;
3. High flexibility;
4. Flatness;
5. Vernisibility in properties directly attributable to materials used.

Unified paper sheets having the above properties are ideal as backings for pressure-sensitive tapes, particularly those used as masking tapes, for example, during the spray painting of automobiles. In such tapes, not only is their unexpectedly high delamination resistance and low cost of importance, but the extraordinary conformability of the backing combined with its flatness and tear strength permit easy application to curved and irregular surfaces and give sharply defined paint lines without feathering at the edges of the tape. Due to its flat backing and to the ease with which the tape may be applied, abrasion of the fingers of the worker applying the tape also is minimized.

Heavier weight unified papers possessing the above properties also are ideal for use in artificial leathers where their superior flexibility, soft hand, freedom from blistering or splitting and freedom from fiber picking are of great importance. Papers according to this invention also are highly desirable as sandpaper backings and when used for this purpose, provide sandpapers possessing superior flexibility, tear strength and crack resistance.

Starting webs according to this invention may be creped or uncreped and include the conventional saturating papers of the prior art. Similarly these webs may be formed from fibers which are substantially straight and possess only natural curl or they may include a substantial proportion of fibers to which a substantial amount of curl or crimp has been imparted prior to web formation. The starting web comprises cellulosic fibers oriented predominantly parallel to the web faces and flatterly assembled in overlapping, crossing relation with one another. When the term “face” is used in referring to the starting web or paper according to this invention, it refers to the outline of one surface of the web, roughly as it would appear to the naked eye. If the web or paper is pictured in a horizontal position, it possesses two faces, i.e., a top face and a bottom face. The “body of the web” or “web body” is that portion of the web or paper between its two faces.

The fibers in the web are relatively long compared to the thickness of the web so that the ratio of the average length of the fibers to the caliper of the web is at least about four to one, preferably at least about ten to one. This means that, even in the most extreme cases, the overall orientation of a given fiber from end to end can only be at a slight angle to the faces of the starting web and that normally this angle is negligible.

The starting web is compacted horizontally while the cellulosic fibers in the web are plasticized with moisture and heat to a temperature sufficient to generate moisture vapor. Compacting forces are applied to the web horizontally with respect to the web faces while the web body is held or restrained against substantial crinking in a vertical direction. In other words, forces are applied approximately perpendicular to the direction of the compaction forces to resist the tendency of the web to crinkle and maintain the web faces substantially parallel to one another. The compacting forces may be applied longitudinally, or in the machine direction of the web, or laterally in the cross machine direction.

The lowest degree of compaction, i.e., percentage of length required for utility in this invention, is not greater than about 5% with some papers. However, with most papers more exhaustive treatment may be desirable. For instance, about 10-25% compaction normally is preferred, particularly when the web is treated in one pass as will be described more fully hereinafter. The upper limit of the degree of compaction is determined both by the nature of raw untreated paper and the application for which the unified paper is intended. In a wide diversity of raw papers intended for various applications, the upper limit of compaction desired might be as high as 40% or more and in some cases approach the lower limit of another paper intended for a different end use.

In general increasing degrees of treatment tend to decrease the tensile strength of the raw paper. However, the physical properties of a paper prior to the incorporation of the unifying material are of secondary importance in determining the usefulness of the unified paper product. The presence of a unifying material, especially in relatively large amounts relative to the untreated fiber, usually results in marked increases in such properties as tensile, tear and elongation as well as in resistance of the sheet to delamination, or splitting.

In general the amount of impregnant incorporated into the paper web is in an amount of about 10-150 percent impregnating solids by weight of the raw paper (dry web), the minimum amount thereof, comprising in all cases, an amount sufficient to unify said web to a degree whereby the forces of adhesion of the particular adhesive coated on the backings are insufficient to cause delamination of an underlying ply of tape, for example. Although the normal practice is to treat a water plasticized web, under certain circumstances the water may comprise only a small percentage of the web weight as it is treated. The desired content of the sheet at the time of treatment is determined both by the nature of the raw paper and the end use contemplated. In general the more dense the untreated paper the greater the water content when compressed if tensile is to be retained in the finished unified paper or tape backing. Thus, a flat paper of fairly dense structure may be most advantageously treated at a water content, limited only by the amount permitted by the equipment and process, that is, the maximum which can be held by the sheet under the conditions of compaction inherent in the process. Normally and preferably the moisture content during treatment should be about 10-20% by weight of the web. However, it may be most advantageous to treat more open sheets, such as an open kraft saturating paper, at a water content at or even below that of equilibrium between the water content of the sheet and normal testing conditions for paper, 50% R.H. at 72° F. Variations of types of paper between these two types may require, or be advantageously treated, at moisture contents varying between these extreme limits. Severe treatment under conditions of little water plasticizing tends toward higher elongation by longitudinal compression and under usual conditions a higher degree of uniformity of the resulting paper. Whatever its moisture content, at least one side of the web is heated during treatment to a temperature sufficient to generate moisture vapor, i.e., at least about 212° F., as will be described more fully hereinafter.

By the compacting operation there is introduced a distortion of the fibers by contracting the area in which they lie, while at the same time holding the faces of the web flat and parallel thus preventing appreciable thinning of the web. Simultaneously, a substantial pressure is maintained on the faces of the web so as to prevent creping of the web and also to force the distorted fibers into voids within the web. Upon drying, the changed orientation of the fibers is retained and the web exhibits firmness and strength from the mutual adhesion of the fibers. When the web is subjected to tensile stress there is an inertia to reformation arising from the mutual adhesion of the almost continuous distribution of the fibers. However, as the tensile stress is increased, the nearly continuous structure of the fibers progressively yields and the web assumes an added width or length approximately equal to the width or length by which the paper web had been compressed. Upon still further increasing the tensile...
stress, the mutual adherence of the fibers which maintains the structure of the web breaks down and the sheet ruptures.

Compaction, longitudinally or laterally, in the manner described, causes the fibers to become compressively dis-6 creased and pushed together within the body of the web. Those fibers which originally are oriented pri-
arily in the direction of compaction are caused to col-
lapse by taking a multiplicity of kinks, crimps or tight
loops as portions of the fibers are forced closer together and into the voids within the web. These kinks, crimps or tight loops, hereinafter called "kinks" occur, not only in a plane web place, but when the fibers are perpendicular thereto so that a large proportion of fiber segments forming the kinks are oriented at an angle to the web faces.

If the starting web is relatively dense as in a flat com-
pact paper, it will retain substantially the same density during compaction and if it is dense and moderately hy-
drated, for instance, fiber-to-fiber bonds will be broken so that its saturability will be increased somewhat during compaction; but if the starting web is relatively open, such as in creped saturating papers, it will be considerably densified and flattened out during compaction.

The impregnation may be incorporated in the web either before or after compaction but impregnation prior to compaction is preferred for three reasons, i.e.,
(a) Greater compaction efficiency is attainable,
(b) Impregnation may be performed in conjunction with compaction for plasticizing the fibers during com-
paction when an aqueous impregnant is employed, and
(c) Better paper properties are attainable with an aqueous impregnant than when the web is first compacted and then impregnated.

Greater compaction efficiency, i.e., actual degree of compaction divided by theoretical degree of com-
paction times one hundred, is attainable when the web is impregnated prior to compaction, especially when compaction occurs when the web is relatively "dry" and at low levels of impregnation. Since it is highly desirable to treat the web at a low moisture content to avoid mois-
ture retention problems, and low levels of impregnation are desired to save cost; it is apparent that improved com-
paction efficiency under these conditions is of great im-
portance.

The starting web may be impregnated with an aqueous impregnant, dried to bring the moisture content to about 10-20% by weight of the web, for instance, and then compounded under the above described conditions with excellent results. In this case the moisture carried into the web with the impregnant acts to plasticize the fibers of the web.

The process of this invention also is highly advan-
tageous because of the variety of cellulosic fibers and impregnants which may be employed. As a result, the papers of this invention are versatile in so far as their properties, which may be attributed directly to the materials employed in their manufacture are concerned. For instance, both aqueous and solvent based impreg-
nants may be used although, when the impregnant is incor-
bated in the web after compaction, solvent based impregnants give better results. However, if the im-
regnant is incorporated prior to compaction, it makes no difference whether it is water based or solvent based.

Thus, it is possible to use the well known and relatively inex-
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dious lattices of the prior art in the process of this invention with maximum results.

In addition, whereas relatively high modulus polymers are not acceptable as unifying agents when applied in the relatively high percentages of unified papers according to the prior art if a flexible paper is desired, such polymers may be highly desired, and in fact preferred, for pressure-sensitive masking tape backing at the lower levels of impregnation made possible by this invention. For instance, polymers in the transition re-
gion of viscoelastic behavior, and even in the upper range of this region, are advantageously employed in unifying papers for certain applications according to this invention when the impregnant incorporated is about 10-50% by weight of the dry fibers. For instance, higher values for delamination resistance and other physical characteristics are attainable with a high modulus polymer when this amount of impregnant is used. The transition region of viscoelastic behavior, referred to above, is described in some detail by Arthur V. Tobolsky, in "Stress Relaxation Studies," at page 65 in Rheology, Theory and Application, volume II, edited by Frederick R. Erich and published by Academic Press Inc., New York, N.Y., in 1958. Such polymers include unplasticized polyvinyl acetate poly-
mers and copolymers, high styrene butadiene-styrene co-
polymers, and the like. Polymers in this region are pre-
ferrred for masking tapes according to this invention to give the tape sufficient firmness for ease in application when only about 10-30% by weight of the unifying im-
regnant is present.

In certain cases it may be advantageous to perform im-
regnation and compaction in more than two steps. For instance, if the starting web is relatively dense, flat and moderately hydrated, it first may be compacted to break some fiber-to-fiber bonds, then impregnated with an elas-
tomeric aqueous latex, and finally compacted again, for optimum results. Or, the starting web may be impreg-
nated with a relatively small amount of an aqueous im-
pregnant prior to compaction, then compacted with maxi-
mum compaction efficiency due to the presence of the im-
pregnant in the web, and finally impregnated with a maxi-
mum amount of a solvent based impregnant, for optimum results.

Other and further advantages of this invention will be
apparent from the following description and claims taken

FIG. 1 is a diagrammatic side elevation of simple appar-
atus useful in performing the compacting step or steps
employed in the invention;

FIG. 2 is a diagrammatic side elevation of apparatus
which may also be employed in conjunction with the
apparatus of FIG. 1;

FIG. 3 is a cross section of an adhesive tape utilizing,
as the backing, a unified sheet treated in accordance with
this invention;

FIG. 4 is a diagrammatic side elevation of somewhat
different apparatus useful in performing the compacting step, or steps, according to this invention;

FIG. 5 is a greatly enlarged schematic view, partly in
section and partly in elevation, of a longitudinal cross
section through a flat, impregnated kraft paper, prior to
compaction according to this invention, at an enlargement
of approximately 400 to 1;

FIG. 6 is a similar greatly enlarged schematic view of a
longitudinal cross section through a creped, impregnated
saturating paper according to this invention and at the
same enlargement as FIG. 5;

FIG. 7 is a schematic view similar to FIGS. 5 and 6,
and at the same enlargement, of the webs of either FIGS.
5 or 6 as they would appear after being compacted accord-
ing to this invention.

The apparatus of FIG. 1 comprises rolls 10, 11, 12 and
a heated driven roll, or drum, 13 and a thick belt 15 with
a contractable surface layer preferably of rubber of durom-
er hardness sufficient to prevent creping of the paper
web. This belt may be formed of natural rubber or rub-
er substitutes, a relatively inextensible layer faced with a
readily contractile and contractable surface layer of any
suitable material of smooth contractile surface of suf-
ficient hardness and extensibility, rolls 10 and 13 being
adjustably movable toward or from each other so as to pro-
perly nip the belt 15 between them where the belt
passes from roll 19 to the drum 13, and spaced away from the drum 13 sufficiently to give the belt a
short straight run from the drum 13 to roll 12. As the
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7 belt passes from roll 10 to the drum 13, the outer surface of the rubber belt which is convexly curved on roll 10 becomes concavely curved on the drum 13 and accordingly shortens. A paper web, which has been impregnated with a water base impregnant, fed in between the belt and the drum 13 where this shortening of the belt surface is taking place, is forced into frictional contact with the contracting surface of the belt that the belt surface tends to compact, or compress, the web, parallel with the surfaces of the web. While the apparatus shown is intended to compact the web in the machine direction of the web, the web may be compacted laterally, i.e. transversely of the direction in which it was made on the paper machine, by turning short lengths sidewise and passing them individually through the apparatus. The drum 13 must be accurately machine ground and finished to a true cylinder of smooth periphery. It is heated not only to cause a partial drying of the web but also to lower the coefficient of friction between the drum and the moist web while at the same time heating the contained water and thus cause a softening and increased flexibility of the fibers. The resulting loss of water in the web makes room between the fibers for further compaction of the web as may be desired. The coefficient of friction between the wet web and the heated drum 13 is most effectively reduced at drum temperatures about 212°F. The coefficient of friction of the surface of the drum 13 is relatively low as compared to the coefficient of friction of the web-containing surface of the belt 15, so that under the influence of the contracting belt surface the paper web tends to partake of some contraction of the belt surface and slide with relation to the roll surface. During the interval, or longitudinal compaction of the paper, the tension in the rubber belt is maintained sufficiently high so that with the hardness of the belt surface the pressure between the rubber belt 15 and the drum 13 prevents the paper from creping and keeps the surfaces of the paper substantially flat and parallel, so that as the web is compressively shortened, individual fibers of the paper which lie generally crosswise or lengthwise of the web or in the direction of shortening are compressively distorted crosswise or lengthwise within the body of the web. Rubber is preferred as the material for the contracting surface, such as that of the belt 15, because of its ability to withstand strong tension and heavy pressure transverse to its surface, thus enabling a relatively large and effectively compacting force of this surface to take place without allowing the paper to crepe in response to such contraction. Rubber is also preferred for its continuously smooth surface and for its ability to grip the paper frictionally to the extent of compacting the paper laterally or longitudinally in the presence of the heavy pressure exerted between the belt and the drum 13 for prevention of creping. In the apparatus as shown, it is preferable that the rubber be reinforced by comparatively inextensible material such as heavy canvas or layers of strong cords so that the necessary high tension in the belt may be maintained and also so that the surface of the belt will expand and contract uniformly while passing over the roller 10 and the heated driving drum 13.

The apparatus of FIG. 2 was employed in conjunction with the apparatus of FIG. 1 principally to facilitate drying of the paper web. As shown in FIG. 2, a moisture-permeable belt 21 passes over an intake roll 21a and thence onto and around a heated drum 20, holding the paper web against the drum during drying.

Certain other mechanisms and procedures useful for carrying out the compression procedure employed in producing the backings of the present invention are shown and described in United States Patent No. 2,021,975 and Australian Patent No. 160,684.

Referring to FIG. 4 of the drawings there is shown apparatus similar to that of FIG. 1 which comprises: a heated metal drum 31 having a smooth, inelastic substantially unyieldable surface 32, a nip roll 33 and a relatively thick continuous rubber compacting belt 34 passing over the nip roll 33 and between the nip roll and the heated drum 31. The compacting belt 34 is similar to the belt shown in FIG. 1 and presents a contractile, yieldably supported rubber outer surface 35 toward the drum. After it leaves the nip roll 33, the belt wraps around a portion of the drum surface 32, say about a 90° segment thereof, leaves the surface of the drum to pass around a tensioning roll 36 spaced to the left of the drum in FIG. 4, and then passes down from the tensioning roll 36 around a bottom guide roll 37 and finally back up to the nip roll 33. The amount that the belt 34 wraps around the drum 31 can be considerably less than a 90° segment of the drum surface or it can be considerably more as evidenced by the approximately 180° wrap shown in FIG. 1.

The nip roll 33 is adjustable toward and away from the surface of the drum 31 to control pressure upon the belt 34 passing between the nip roll and the drum and thereby regulate the pressure between the drum 31 and the belt 34 at the nip 35. The tensioning roll 36 is considerably spaced from the drum so that the belt passes from the drum to the tensioning roll in a relatively taut condition. The tensioning roll 36 also is adjustable toward and away from the drum 31 to regulate the tension on the continuous compacting belt 34 and thereby regulate the pressure between the drum and the belt after the belt leaves the nip 35.

A starting web 40, which may have a suitable impregnant or unifying material already incorporated therein, is unwound from the supply roll 41 at the right of FIG. 4 and led into a steam chest 42 wherein it is exposed to saturated steam for a period of approximately 30 seconds to plasticize the web and adjust its moisture content to about 10-20%, preferably about 15-20% by weight of the dry web. The moistened web 40 is led from the steam chest over an exit roller 43 and over a pair of guide rollers 44 and 45 and then down into the nip 35 formed between the drum 31 and the belt 34 where the belt passes over the nip roll 33.

As the belt 34 passes over the nip roll 33, its outer surface 35 follows a convex path while its inner surface follows a concave path with respect to the axis of curvature of the belt with the result that its outer surface 35 is stretched, or expanded, as at A in FIG. 4. Correspondingly, as the belt 34 passes the nip 35 and begins to follow the surface 32 of the drum 31 instead of the surface of the nip roll 33, its outer surface 35 begins to follow a concave path and accordingly shortens, or contracts, as at V in FIG. 4. The belt is compressed somewhat at the nip 35 so that it normally contacts the drum 31 and is spaced above the plane P=P common to the axes of the drum and the nip roll while its outer surface 35 still is expanded in its convex path over the nip roll 33. Thus, the outer surface 35 of the belt 34 is expanded where it first contacts the web 40 and where the web 40 first is gripped between the belt 34 and the drum 31. In the area where the belt 34 is compressed due to the pressure between the drum 31 and the nip roll 33 its outer surface 35 tends to remain expanded to some extent even after it passes the center of the nip 38 and normally would contract as it starts to follow its concave path around the drum 31. However, as soon as the expanding influence of the nip is passed, the outer surface 35 of the belt contracts as the belt approaches its normal thickness and completes passage from its convex path around the nip 35 to its concave path around the drum 31.

As indicated in connection with FIG. 1, the surface 32 of the drum 31 is quite present and the drum is heated to further plasticize the web 40 and reduce the coefficient of friction between the drum and the web. In addition, the heated drum 31 serves to dry the web to some extent as it passes between the belt 34 and the drum after leaving the nip 38. Preferably, the inter-face between the web 40 and the surface 32 of the drum
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is heated to a temperature of at least about the boiling point of water, i.e., 212° F., so as to generate moisture vapor. This moisture vapor both plasticizes the web 40 and creates a thin film or cushion of steam which materially reduces the coefficient of friction between the wet web and the heated drum. On the other hand, the web-contacting surface 35 of the belt preferably is relatively rough compared to that of the drum and the natural coefficient of friction of the belt is therefore greater than that of the drum. Thus, because of these differences between the coefficients of friction of the surface 32 of the drum and the surface 35 of the belt and because of the layer of steam between the web 40 and the drum, the paper web 40 tends to stick to the belt 34 and slip on the drum 31 with the result that when the outer surface 35 of the belt contracts, the web 40 is compacted horizontally or parallel to its median plane, in the direction of contraction of the belt surface. Since the belt contracts longitudinally, or in the machine direction, the web also is compacted longitudinally. During compaction the web 40 is compacted between the belt 34 and the drum 31 by the pressure at the nip 38 and the tension on the belt 34 in such a way that the belt 34 exerts sufficient pressure with respect to the web faces to prevent substantial wrinkling of the body of the web 40 and hold the web faces substantially flat and parallel to each other. Normally, for a belt approximately 12 inches wide commercially in use, the nip force is in the neighborhood of about 150-250 pounds per inch of width and the tension on the belt is in the neighborhood of 120 pounds per inch of width. Normally, the web 40 is compacted longitudinally by about 10-25% of its original length by passing the web once through the apparatus of FIG. 4 in the machine direction so described. It is preferred that the impregnate be incorporated in the web 40 prior to compaction for the reasons indicated hereinafter, particularly because higher compaction efficiencies can be attained. This improvement in efficiency of compaction which occurs when the web is impregnated prior to compaction may be explained on the theory that the impregnate exerts a "clutch action" upon the outer surface 35 of the contractible rubber belt 34, or grips the belt better, thereby increasing the frictional resistance of the web 40 to any movement relative to the outer belt surface 35.

For highest compaction efficiency, it is preferred that the temperature of the surface 32 of the drum be in the neighborhood of about 250° F. It also is preferred that the contractible belt 34 be cooled below the temperature of the drum particularly when the drum temperature is in the neighborhood of 250° F., to further increase the grip between the web 40 and the belt 34 and increase the effective life of the belt.

Referring to FIG. 4, a first cooling water spray nozzle 48 is positioned inside the belt 34, after the belt leaves the drum 31 in such a way as to direct a spray 47 of cooling water upon the underside of the belt before it reaches the tensioning roll 36. A second spray nozzle 48 is positioned outside the belt 34 opposite the tensioning roll 36 in such a way as to direct a spray 49 of cooling water upon the belt's outside surface 35. Finally, a bath 51 of cooling water is located in a trough 52 underneath the drum 31 and the bottom guide roll 37 is positioned about 1/2 inch partially submerged in the bath 51 so that the belt 34 passes through the bath and is cooled thereby. After leaving the bath 51 the belt 34 passes through the nip formed by a pair of squeeze rollers 52 which are adjusted to squeeze all but minute traces of the cooling water from the belt. When the surface of the drum 31 is maintained at about 250° F., the belt 34 preferably is maintained at about 170° F. by the cooling apparatus described. In this way, the belt has a much longer useful life than it would have if operated at the temperature of the drum.

After the compacted and partially dried web 40 leaves the surface 32 of the drum 31, it passes around a guide roller 53 and then down to a series of positioning rollers

54 which hold the web 40 out flat as it passes over a radiant heater 55 which completes the drying operation. The web 40, now in the form of a unified paper according to this invention, finally is led over another guide roller 56 and onto a wind up roll 57 on which it conveniently may be stored or supported for further processing.

FIGS. 5-7 are schematic drawings of longitudinal cross sections of portions of illustrative papers according to this invention before and after compaction. These cross sections are taken along lines extending longitudinally in the direction of compaction, of the web. Each of the papers, shown, contains the desired unifying amount of impregnant but for the sake of clarity the impregnant is shown only at the left end of each of the figures. The distribution of impregnant at the ends of the figures is typical of impregnant distribution throughout the body of the webs at the level of impregnation shown. In each figure, the enlargement is about 400 to 1. The dotted lines extend along the top and bottom faces of the webs represent the web faces, i.e., the surfaces of the webs as they would appear to the naked eye or to the center-lines designated M—P represent the median planes of the webs.

FIG. 5 depicts an impregnated but uncompacted flat starting web such as that designated F—61 Flat Impreg. Paper, in Example I hereinafter. This paper is formed from relatively unbeaten, softwood fibers 60 and is impregnated with particles or film layers 74 of a conventional rubbery latex, such as a unifying agent comprising a butadiene-acrylonitrile copolymer modified with a minor amount of a butadiene-styrene copolymer and a small amount of a phenolic resin, as will be described more fully hereinafter in Example I. The fibers 60 are substantially straight and possess only their natural curl and are oriented in the web predominantly parallel to the web faces 62 and flatly assembled in overlapping, crossing relation with one another extending in various directions parallel to the median plane M—P of the web. The fibers 60 are relatively long compared to the thickness of the web, i.e., averaging about 3.5 millimeters or 130 mils in length whereas the average thickness of the paper is about 4 or 4½ mils. It is obvious from this that the fibers 60 themselves lie flat in the web so that the overall orientation of a given fiber from end to end can only be at a slight or negligible angle to the facings of the starting web.

FIG. 6 depicts a creped starting web formed from the same stock as the paper of FIG. 5 and impregnated with the same amount of the same impregnant 61. This paper is typical of that designated F—61 Crepe, Impreg. Paper, in Example I hereinafter. As a result of creping the web has been opened up by movement of overlying fibers 60 in the web away from one another to increase the size of the voids or interstices 63 between the fibers. The web comprises large undulations, crepes or wrinkles which are not clearly identifiable in FIG. 6 because of their size. In other words, the tip 64 of only one of these undulations appears in FIG. 6. Crepped papers of this type are most open adjacent to the tips of these undulations, or crepes, and it is one of these most open portions of the web which is illustrated in FIG. 6. To show the whole of this undulation in the body of the web, it itself, it would be necessary to have drawing about 3, 4, or 5 times as long as that of FIG. 6. As explained in connection with the paper shown in FIG. 5, the fibers 60 in the paper, or web, of FIG. 6 are relatively long compared to the thickness of the web and flatly assembled in overlapping crossing relation with one another in the web.

FIG. 7 depicts the impregnated webs of FIG. 5 and FIG. 6, after compaction. This paper is typical of those designated F—61 Flat, Compacted 1, Web, and F—61 Crepe, Compacted 1, Web, in Example I hereinafter. During compaction in apparatus such as shown in FIG. 4, for example, the webs of FIGS. 5 and 6 are held between the belt 34 and the drum 31 and the belt applies sufficient pressure to the web acting perpendicu-
larly with respect to the web faces 62 to prevent substantial wrinkling of the web body and hold the web faces substantially flat while in contact with the drum and the belt.

The fibers 60 in the web of FIG. 7 are compressively distorted longitudinally, crowded and pushed together within the body of the web. The fibers, originally oriented primarily in the direction of compaction, i.e., longitudinally, of the web, comprise kinks, or crimps, 65 which show in the plane of the cross section. This plane is perpendicular to the web faces 62 so that the fiber segments 66 forming these kinks are oriented at an angle to the web faces 62. It appears from FIG. 7 that a large portion of the fiber segments 66 forming the kinks 65 are oriented at an angle to the web faces 62. As in FIGS. 5 and 6, the sectioned portions 60 of the fibers 60, represent fiber segments which are oriented, at least in part, laterally in the web, i.e., transversely of the direction of compaction. Even though certain of the kinks 65 formed in the fibers as a result of compaction are reflected in small crenulations, i.e., very small and closely spaced humps 67 which may be visible to the naked eye, and adjacent depressions 67a in the surfaces of the web, the web body, itself, is not substantially wrinkled. During compaction, the web body is densified, i.e., its fibers are brought closer together, and the web is maintained substantially flat. According to this invention, a large number of closely spaced kinks, or crimps 65, some of which are relatively tight, may be formed in the fibers originally extending in the direction of compaction. For instance, normally about 100-400 kinks per inch are formed in the web in the fibers originally extending in the direction of compaction. This means that there may be about 12-48 kinks per fiber extending in this direction for papers formed from long fibered softwood pulps such as used in the free and open saturating papers of the prior art.

While in general the faces 62 of the web remain substantially flat, it should be noted that the top face 62, in FIG. 7, representing the face next to the compacting belt 34, is relatively rough or bumpy compared to the bottom face 62, representing that next to the drum 31, which is comparatively smooth. Thus, the faces 62 of the web tend to correspond somewhat with the respective surfaces of the belt 34 and the drum 31 with which they have been in contact during compaction.

In undried papers according to this invention, as illustrated in FIG. 7, the fibers 60 are extensively bonded together in their crowded and distorted state by the unifying material, or impregnant 61, in the web. The impregnant forms bonds between fiber segments 66 making up the kinks and adjacent fibers in the web, which bonds tend to retain the kinks so that the bonded fiber segments 66 tend to remain oriented at an angle to the web faces 62 and thereby resist delamination, or laminar separation of the fibers in the paper.

As indicated above, while compaction, according to this invention, does not result in substantial wrinkling, i.e., the formation of relatively large undulations in the body of the web, itself, it may result in the formation of crenulations, or very small and closely spaced humps, in one or both of the surfaces of the web but normally in the web surface in contact with the relatively rough surface of the compacting belt. However, in general the web faces of the resulting undried compacted paper, according to this invention, are substantially flat and in all such papers the fibers are compressively distorted, crowded and pushed together within the body of the web and the fibers originally oriented primarily in the direction of compaction comprise a multiplicity of kinks, i.e., in the order of 100-400 kinks per inch in the web, with a large proportion of the fiber segments forming the kinks oriented at an angle to the web faces.

While the apparatus of FIG. 4 has been described in connection with the treatment of paper which is impregnated prior to compaction, it also can be used for treatment of paper which is intended to be impregnated after compaction. Also, as mentioned hereinbefore and as will be described in the following examples, the starting web may be impregnated just prior to compaction so that some of the moisture included during impregnation may be used to plasticize the web and the steam chest may be eliminated.

The following additional examples are included to illustrate various embodiments of the process and products according to this invention.

**EXAMPLE I**

A conventional saturating grade creped paper formed from relatively unbeaten, bleached, softwood, kraft fibers is impregnated with a conventional low modulus water based rubbery latex of the composition, set forth below, and having about 27% total solids, by passing the web through a bath of the impregnant composition and running the web through conventional squeegee rollers; and the impregnated web that is dried by conventional means. Impregnation is controlled so that the impregnant add-on, after drying, is about 25% by weight of the dry web.

### Aqueous impregnant composition

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade name</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butadiene-acrylonitrile</td>
<td>Goodrich Hiyear OR-20</td>
<td>70%</td>
</tr>
<tr>
<td>Butadiene-styrene</td>
<td>Dewey &amp; Almy Dares 621L</td>
<td>15%</td>
</tr>
<tr>
<td>Acrylonitrile</td>
<td>Dewey &amp; Almy Dares 623L</td>
<td>15%</td>
</tr>
<tr>
<td>Heat curing phenol-formaldehyde</td>
<td>Durcal Plates 14,760</td>
<td>13%</td>
</tr>
<tr>
<td>Tetra sodium salt of ethylenediamine</td>
<td>3,147</td>
<td>7%</td>
</tr>
<tr>
<td>Butane-2,3-diol-tetraceteicacid</td>
<td>Nalgeneck Anilox Disper-</td>
<td>6.5%</td>
</tr>
<tr>
<td>Antioxidant</td>
<td>(50)</td>
<td></td>
</tr>
<tr>
<td>Water</td>
<td>(55)</td>
<td></td>
</tr>
</tbody>
</table>

The impregnated web then is compacted in apparatus of the type shown in FIG. 4 essentially as described hereinbefore. The web is steamed to adjust its moisture content prior to compaction to about 15-20% by weight of the dry impregnated web and is compacted at a drum temperature of about 250°F. and a belt temperature of about 170°F. with a belt tension of about 120 pounds per inch of width and a nip force of about 230 pounds per inch of width. The impregnated web is dried by conventional means, by subjecting the web to a temperature of about 350°F. in an air circulating oven for about one minute.

The resulting impregnated paper possesses unexpectedly high delamination resistance for the small amount of impregnant it contains, extraordinary conformability, high flexibility, flatness, high tear strength, good tensile strength, and various other properties which make it particularly suitable for a backing for a pressure-sensitive adhesive masking tape as well as other uses. At only about 25% impregnation, according to this example, great cost savings are realized, yet superior results are obtained. The unexpectedly high delamination resistance of this paper and its importance will be described more fully hereinafter in connection with the data set forth in Table II. For purposes of comparison in analyzing delamination resistance, an additional sample is made and tested by compacting the starting web of this example without impregnant.
In other words, a control sample is prepared which is treated exactly the same as the above sample according to this invention except that the impregnation step is eliminated.

Full data for the original starting web, the control, the impregnated starting web and the resulting unified paper according to this invention, designated "Com- 
pacted I. Web," on tensile strength, tear strength and elongation-to-break in the machine and cross directions; delamination resistance; caliper; basis weight; air porosity; apparent density and bulk; are given in Table 1 below under the heading F-61 Crepe.

A flat starting web is the same as the above-described creped starting web, except for the fact that it is uncreped, is treated in the same way as the creped starting web to provide a unified paper according to this invention which possesses unexpectedly high delamination resistance, conformability, flexibility and elongation in the same order of magnitude as the creped starting web, but higher tensile strength, lower tear strength and lower porosity. Full data for this impregnated and compacted flat starting web and its unimpregnated compacted control are given in Table 1 below under the heading F-61 Flat.

The data on tensile strength and percent elongation given in Table I are obtained by testing the papers in accordance with ASTM, Standard Test D-1000, the elongation figures being percent elongation-to-break, as mentioned above. Tear is determined by a modified Finn test of the type which has become standard in the paper masking tape industry. Delamination resistance is measured by preparing samples and testing them as described by Dunlap in "Some Factors Affecting Ply Adhesion in Latex Saturated Papers," appearing in Tappi, vol. 40, No. 8, August 1957, page 677. Caliper is measured in a conventional manner and basis weight is expressed in pounds per 320 square yards. Air porosity is expressed in seconds and represents the time in seconds required for 400 cc. of air to pass through a paper web placed in a model 4110 Gurley Densometer with conditions controlled in accordance with Tappi Specification No. T-460, M-49. The figures given for apparent density represent the basis weight divided by thickness in mills, and bulk is expressed in cubic centimeters per gram as indicated.

Inspection of the delamination resistance given for the unified papers formed from the flat and creped starting webs of this example, shows the unexpectedly high delamination resistance of these papers compared with the delamination resistance of the impregnated starting webs, for example. The delamination resistance of the control, i.e., the compacted unimpregnated starting web, also is higher than the unimpregnated starting web. This, in itself, is surprising since prior art and prior experience indicate that compressive treatment of the web, if anything, may decrease delamination resistance. For instance, Cluett in United States Patent No. 2,624,245, which described a compacting operation similar to applicant's and essentially the same as disclosed in United States Patent No. 2,021,975 and Australian Patent No. 160,684, referred to hereinbefore, indicates that his compacting operation decreases the tensile strength of his paper. (Col. 6, lines 56 and lines 34-55.) All things being equal, a decrease in tensile strength of an unimpregnated web normally is associated with a decrease in delamination resistance. Thus the increased delamination resistance of the control samples is surprising in itself.

However, even if it is assumed that it would not be unexpected if the increased delamination resistance of a particular unified paper according to this invention merely equaled the sum of:

(a) The delamination resistance of the impregnated uncompact ed starting web, and

(b) The increment representing the increase in delamination resistance attributable to compaction alone, i.e., the difference between the delamination resistances of the compacted control and the unimpregnated starting web, the delamination resistance of unified papers according to this invention is unexpectedly high as exceeds the above sum by an appreciable amount and therefore clearly exhibits synergism.

This now will be explained in more detail with particular references to Table II below. From theoretical considerations, it will be apparent that the total delamination resistance (D_P) of any given "united" and/or calendered or compacted paper may be represented by the equation:

\[ D_P = D_T + D_C + D_I + K \]  

(Equation I)

where:

\( D_T \) = the delamination resistance of the untreated paper sheet.

\( D_C \) = the increment in delamination resistance (over \( D_T \)) contributed by the compacting step per se, in the absence of impregnation.

\( D_I \) = the increment in delamination resistance (over \( D_C \)) contributed by the impregnation step per se (in absence of any compaction step).

\( K \) = a constant for any specific set of conditions.

Transposing the above equation to solve for the constant \( K \):

\[ K = D_T - D_T - D_C - D_I \]  

(Equation II)

From Equation II it follows that:

(1) If \( K \) is zero, then the two operations mentioned above are purely additive or cumulative in their effect on the delamination resistance of the finished product;

(2) If \( K \) is a negative number, the two operations are antagonistic in their effect on delamination resistance of the finished product;

(3) If \( K \) is a positive number, the two operations are synergistic in their effect on delamination resistance of the finished product;

(4) In those instances where the two operations produce a synergistic (or an antagonistic) effect, the magnitude of the synergistic (or antagonistic) effect (expressed in terms of a percentage of the delamination resistance of an equivalently impregnated and uncompressed paper sheet) is given by the equation:

\[ \text{Percent synergism (or antagonism)} = \frac{100 K}{D_T + D_I} \]  

(5) If the basis of comparison is an equivalently compressed but unimpregnated sheet, the percent synergism (or antagonism) is given by the equation:

\[ \text{Percent synergism (or antagonism)} = \frac{100 K}{D_T + D_C} \]

Table II tabulates the results obtained by applying the foregoing fundamental principles to some of the papers of Table I including the unified papers of this example.

EXAMPLE II

Both the flat and creped unified papers of Example I are knife coated, in sheet form, with the following backsize composition, or release agent, diluted in a (50:50)
solution of toluene and methyl-ethyl-ketone to about 31 percent total solids:

**Backsize Composition**

**Material:**  
Parts by weight

| Epoxy resin | 6 |
| Polyvinyl chloride-acetate (87:13) copolymer | 600 |
| High molecular weight polyester (plasticizer) | 210 |
| TiO₂ | 150 |
| 50% solution | 240 |

**318 Solution**

**Material:**  
Amount

| Sorbitan monostearate | lbs | 60 |
| Toluene dioctylamide | lbs | 15 |
| Terephthalamine | 5 |
| Toluene | gal | 31/2 |

The coated paper is exposed to a temperature of 300° F. for approximately 1 minute to cure the backsize. The resulting backsize coating weighs about 0.25 ounces per square yard. The opposite sides of the resulting coated sheets next are reverse roller coated, with the following pressure-sensitive adhesive mass dissolved in toluene to about 35% solids. The coated mass is dried and then cured by exposing the sheets to a temperature of 390° F. for approximately 1 minute. The resulting pressure-sensitive adhesive layer weighs about 2 ounces per square yard.

**Adhesive Mass**

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade name</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyisoprene</td>
<td>Pale Crepe #1</td>
<td>20.0</td>
</tr>
<tr>
<td>Polyethylene (70) copolymer</td>
<td>GRS 802</td>
<td>10.0</td>
</tr>
</tbody>
</table>
| Diethylene glycol ester of dehydro-  
  pherolic acid |  |  |
| p-Octylphenyl hemisalicydehyde | Amberol S137 | 19.0 |
| Zinc stearate | Prestol | 25.2 |
| 2,5 dibenzyl-2,4 hydroquinone | Rontovar A | 2.2 |
| Terephthalic acid | Refined Products | 1.6 |
| 2,5 dibenzyl-2,4 cresol | Terephthalic Acid | 2.2 |

Each of the resulting sheets, coated on one side with the backsize composition and on the other with the pressure-sensitive adhesive layer, is then slit into narrow widths by techniques which are conventional in the paper industry and the resulting pressure-sensitive adhesive tape is wound in successive helical convolutions upon itself in the form of a roll with the pressure-sensitive layer facing inward. A piece of tape recording to this example is shown in FIG. 3 wherein a layer 2 of a normally tacky and pressure-sensitive adhesive is applied to one side of a unified sheet 1 produced according to this invention and the backsize, not shown, is applied to the other side of the sheet 1.

The resulting rolls of tape formed from the flat and the creped starting webs of Example I are rapidly windable without any sign of delamination of the paper backing and are capable of being applied to smooth and rough surfaces and removed therefrom without delamination. After unwinding, both of these tapes possess a relatively high elongation, i.e., over 25% and are quite flexible. More importantly, perhaps, these tapes possess extraordinary ability to be conformable smoothly and easily to curved surfaces and other three-dimensional objects and to be smoothly applied in different shapes to flat surfaces. For instance, they may be applied in relatively sharp arcs to a flat surface without wrinkling of one edge of tape and when used as a masking tape, give sharply defined paint lines without feathering at the edges of the tape. This is due mainly to the fact that the tape lies flat upon the surface to be painted and is not crinkled at its edges. The flatness of the tape backing also facil-

ates application of the tape in the first place and minimizes abrasion of the fingers of the worker applying it. In addition, these tapes are quite inexpensive, mainly due to the small amount of impregnant contained in the backing and the great variety of less expensive papers which may be used.

**EXAMPLE III**

A unified paper according to this invention is made by impregnating the creped starting web of Example I with the following solvent based impregnant composition, in the manner described in Example I, to provide an impregnant add-on after drying of about 22% by weight of the dry web. The following materials are dissolved in about 17 gallons of toluene:

**Solvent Impregnant**

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade Name</th>
<th>Lbs</th>
</tr>
</thead>
</table>
| Glycerol ester of hydrogenated \n  resin | Hercules Staybrite Ester  
  E10 | 24.4 |
| Zinc resinate | Newpark Industries Zinc | 2.25 |
| 2,5 dibenzyl-2,4 phenolic resin | Shell Alkyd | 0.75 |
| p-Octyl phenol Formaldehyde | Rohm and Haas Amber | 75.0 |
| 2,5 dibenzyl-2,4 hydroquinone | Monsanto Santovar A-22 | 0.75 |
| Nat. crimp rubber in depolymer-  
  ized form | DPR-Hich Vis.-No. 1 | 75.0 |
| DPR Pigmented Com-  
  pound |  | 9.0 |

The impregnated web then is compacted and dried as described in Example I. The paper then is cured by exposing it to a temperature of about 425° F. in an air circulating oven for a period of about 3 minutes to provide a unified paper according to this invention possessing unexpectedly high delamination resistance, extraordinary conformability, high flexibility, flatness, high tear strength, good tensile strength and the like as described in Example I. As in Example I, full data are given on these and other properties for the unimpregnated but compacted control, the impregnated web and the resulting unified paper in Table I below under the heading F-61 Crepe (Solvent Impreg.). Data for the starting web are the same as in Example I opposite F-61 Crepe. These data are obtained in the manner described in Example I. Data also are given for this paper in Table II on the synergistic increase in delamination resistance according to this invention.

The unified paper of this example is made into a tape wound upon itself in roll form by coating the paper with a backsize composition and an adhesive mass as described in Example II. The resulting pressure-sensitive adhesive tape is found to have all of the properties described in connection with the tape of Example II and therefore to possess particular advantages for use as a masking tape for curved surfaces and for masking difficult designs.

**EXAMPLE IV**

A unified paper according to this invention is formed from the same creped starting web and the same solvent based impregnant and in the manner described in Example III, but this time the starting web is compacted prior to incorporation of the impregnant, to provide a unified paper which possesses properties which are essentially equivalent to the properties of the unified paper of Example III. These properties are set forth in Table I under the heading F-61 Crepe (Solvent Impreg.) opposite "Impreg. Control." Again, synergism is demonstrated by comparing these results for the paper in Table II. A pressure-sensitive adhesive tape wound upon itself in roll form is fabricated as described for data in Table II and III from the unified paper of this example with equivalent results.
TABLE I

<table>
<thead>
<tr>
<th>Ex.</th>
<th>Paper</th>
<th>Web</th>
<th>Degree of compaction</th>
<th>Lbs. tensile</th>
<th>Lbs. tear</th>
<th>Percent elongation</th>
<th>Ounces</th>
<th>Caliper, mils</th>
<th>Lbs. basis weight</th>
<th>Seconds caliper</th>
<th>A.D.</th>
<th>Bulk e.e.</th>
<th>g/m.</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>MD</td>
<td>XD</td>
<td>MD</td>
<td>XD</td>
<td>MD</td>
<td>XD</td>
<td>MD</td>
<td>XD</td>
<td>MD</td>
<td>XD</td>
<td>MD</td>
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<tr>
<td>I.</td>
<td>8-4 flat kraft...</td>
<td>Starting web</td>
<td>27.5</td>
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<td>96</td>
<td>101.3</td>
<td>2.3</td>
<td>4.2</td>
<td>8.0</td>
<td>5.5</td>
<td>4.2</td>
<td>4.2</td>
<td>31.2</td>
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<tr>
<td></td>
<td></td>
<td>Control</td>
<td>25</td>
<td>6.83</td>
<td>6.8</td>
<td>128</td>
<td>165.4</td>
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<td>6.1</td>
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<td>7.5</td>
<td>8.5</td>
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<tr>
<td></td>
<td></td>
<td>Impreg. starting web</td>
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<td>Impreg. starting web, I</td>
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<td>24.8</td>
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<td>6.5</td>
<td>7</td>
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<td></td>
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<td>Compressed I</td>
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<td>II.</td>
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<td>9.1</td>
<td>19.0</td>
<td>32.0</td>
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<tr>
<td></td>
<td></td>
<td>Control</td>
<td>26.6</td>
<td>5.7</td>
<td>8.3</td>
<td>128.3</td>
<td>22.0</td>
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<td>7.5</td>
<td>6.5</td>
<td>35.5</td>
<td>191.5</td>
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<tr>
<td></td>
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<td>Impreg. starting web</td>
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<td>33.9</td>
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<td>11.7</td>
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<tr>
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<td></td>
<td>Impreg. starting web, I</td>
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<td>28</td>
<td>12.8</td>
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<td>Compressed I</td>
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<td>14.3</td>
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<td></td>
<td></td>
<td>Control</td>
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<td>6.6</td>
<td>7.8</td>
<td>128.3</td>
<td>32.3</td>
<td>4.9</td>
<td>21.0</td>
<td>7.5</td>
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<tr>
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<td>Impreg. starting web, I</td>
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<td>35</td>
<td>6.5</td>
<td>7.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Compressed I</td>
<td>23</td>
<td>24</td>
<td>12.2</td>
<td>15</td>
<td>123.3</td>
<td>35</td>
<td>7</td>
<td>32</td>
<td>6.5</td>
<td>7</td>
<td>48.5</td>
</tr>
<tr>
<td>V.</td>
<td>Patterson 556...</td>
<td>Starting web</td>
<td>24.6</td>
<td>17.0</td>
<td>64</td>
<td>58.3</td>
<td>1.67</td>
<td>2.8</td>
<td>12.0</td>
<td>2.75</td>
<td>3</td>
<td>29.8</td>
<td>129.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Control</td>
<td>24</td>
<td>15.7</td>
<td>12.3</td>
<td>68.2</td>
<td>108.6</td>
<td>31.1</td>
<td>5.0</td>
<td>22.0</td>
<td>7.5</td>
<td>6.5</td>
<td>35.4</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impreg. starting web</td>
<td>24</td>
<td>14</td>
<td>14.2</td>
<td>68.2</td>
<td>64</td>
<td>1.9</td>
<td>34</td>
<td>7.5</td>
<td>6.5</td>
<td>35.4</td>
<td>1287.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impreg. starting web, I</td>
<td>25</td>
<td>20</td>
<td>17.6</td>
<td>10.3</td>
<td>64</td>
<td>30</td>
<td>5.6</td>
<td>7</td>
<td>9</td>
<td>62.7</td>
<td>&gt;12,000</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Compressed I</td>
<td>24</td>
<td>30</td>
<td>17.6</td>
<td>14.7</td>
<td>94</td>
<td>90.6</td>
<td>17.5</td>
<td>4</td>
<td>40</td>
<td>5.5</td>
<td>7.5</td>
</tr>
<tr>
<td>VI.</td>
<td>Patterson 556...</td>
<td>Starting web</td>
<td>11.9</td>
<td>5</td>
<td>7</td>
<td>74.0</td>
<td>96</td>
<td>9.5</td>
<td>5.0</td>
<td>20.0</td>
<td>7.5</td>
<td>6.5</td>
<td>30.8</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Control</td>
<td>24</td>
<td>10.8</td>
<td>26.5</td>
<td>128.3</td>
<td>22.0</td>
<td>7.5</td>
<td>19.7</td>
<td>7.5</td>
<td>6.5</td>
<td>30.8</td>
<td>181</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impreg. starting web</td>
<td>26</td>
<td>26.3</td>
<td>10.6</td>
<td>90.3</td>
<td>90</td>
<td>10.5</td>
<td>30</td>
<td>8.5</td>
<td>9.5</td>
<td>29.8</td>
<td>178.5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Impreg. starting web, I</td>
<td>26</td>
<td>26</td>
<td>16</td>
<td>11</td>
<td>112</td>
<td>42.8</td>
<td>17.6</td>
<td>8</td>
<td>42</td>
<td>8.5</td>
<td>5.5</td>
</tr>
<tr>
<td>VII.</td>
<td>8-60 crepe...</td>
<td>Starting web</td>
<td>24</td>
<td>30</td>
<td>15</td>
<td>11</td>
<td>112</td>
<td>42.8</td>
<td>17.6</td>
<td>8</td>
<td>42</td>
<td>8.5</td>
<td>5.5</td>
</tr>
</tbody>
</table>

TABLE II

<table>
<thead>
<tr>
<th>Example</th>
<th>Synergism in delamination resistance</th>
<th>Magnitude of synergistic effect, expressed as a percentage of delamination resistance of—</th>
</tr>
</thead>
<tbody>
<tr>
<td>I.</td>
<td>8-4 flat, starting web</td>
<td>ACTION</td>
</tr>
<tr>
<td>II.</td>
<td>8-4 crepe, starting web</td>
<td>ACTION</td>
</tr>
<tr>
<td>IV.</td>
<td>8-4 crepe (solvent impreg.)</td>
<td>ACTION</td>
</tr>
</tbody>
</table>

EXAMPLE V

A relatively dense flat paper starting web, such as sold under the name Patterson 556 by the Patterson Parchment Paper Co., and formed predominantly from moderately hydrated softwood fibers is impregnated with the aqueous impregnant composition of Example I and heated to dry the web as described in Example I, to provide an impregnant add-on of about 20% by weight of the dry web. The resulting impregnated sheet then is compacted as described in Example I at an initial moisture content of about 20% by weight of the dry web, dried and cured as described in Example I to provide a unified paper according to this invention. This paper possesses exceptionally good delamination resistance for the amount of impregnant it contains, i.e., 50 ounces delamination resistance for 20% impregnant. As in the papers of the preceding examples, conformability, elongation and flexibility are unusually good. Tenacity strength is somewhat lower than that of the starting web but still very adequate, while the tear strength is substantially unchanged. The caliper of the unified paper is substantially increased over that of the starting web indicating that embrittlement occurs along the surface of the paper are formed to a high degree. However, the faces of the paper are relatively flat and are capable of being flat against any surface to which they may be applied. Thus, paper according to this example is also particularly suitable as a backing for a pressure-sensitive adhesive tape, such as a masking tape. Full data for paper according to this example are set forth in Table I under the heading Patterson 556, opposite "Compacted 42 I. Web." Similar data are given in this table for the unimpregnated starting web, the impregnated starting web, and for a control in the form of the compacted starting web.

EXAMPLE VI

A unified paper according to this invention which is particularly suitable for a tape backing is formed from the same starting web, the same impregnant and in the same manner as described in Example V with the single exception that the web is compacted before it is impregnated. The resulting unified paper is substantially equivalent to that of Example V, except that its delamination resistance and elongation are not as high in proportion to the amount of impregnant added.

EXAMPLE VII

A saturating grade crepe paper formed from Canadian softwood kraft pulp with a high Alpha cellulose content and comprising a major percentage of fibers which have curls, or kinks, artificially imparted to them by a process similar to that described in United States Patent No. 2,516,384; is impregnated with the aqueous impregnant of Example I and partially dried to bring the moisture content down to about 20%. The sheet, with this moisture content, then is compacted as described in Example I and finally completely dried and cured to provide a unified paper according to this invention having an impregnant add-on of approximately 30%. Detailed data for
this paper and its "control" are given in Table I under the heading F–60 Crepe. The resulting paper possesses excellent delamination resistance, conformability, elongation, flexibility, flatness, and is particularly suited for use as a paper masking tape backing.

EXAMPLE VIII

A 400 yard length of a cheap (15¢/lb.) toweling grade, absorbent paper of 36# basis weight, produced by the

Brown Co. and sold primarily to the diary industry under the Brown trademark "Kowiol," is wound upon a core and stored for about two weeks time at room temperature and coded "K" for identification purposes. Another 400 yard sample of the same starting web is impregnated with an aqueous latex to the extent of about 15% of its original weight as described in Example I; the latex composition used being as follows:

TABLE A

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade name</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butadiene-acrylonitrile</td>
<td>Goodrich &quot;Hyco Or-26&quot;.</td>
<td>3,320</td>
</tr>
<tr>
<td>copolymer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Butadiene-styrene (50:50)</td>
<td>Nangstuck butadiene styrene copolymer 5633</td>
<td>3,320</td>
</tr>
<tr>
<td>copolymer</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Heat curing phenolinformaldehyde resin</td>
<td>Durco phenolinformaldehyde resin No. 15/50, Enfield Products-&quot;Permaflex 850.&quot;</td>
<td>81.6</td>
</tr>
<tr>
<td>Tetra sodium salt of ethylene diamine tetraacetic acid</td>
<td></td>
<td>40</td>
</tr>
<tr>
<td>Water</td>
<td></td>
<td>11.360</td>
</tr>
</tbody>
</table>

The impregnated 400 yard sample is dried and stored at room temperature for two to three weeks, wound upon a core and coded "L" for identification purposes. Each of the foregoing 400 yard samples ("K" and "L") then is compacted as described in connection with FIG. 4 under the conditions set forth in Table B below:

TABLE B

<table>
<thead>
<tr>
<th>Nip roll:</th>
<th>Approximate 230 pounds per inch of width.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter</td>
<td>6 inches</td>
</tr>
<tr>
<td>Rubber belt:</td>
<td>1.25 inches</td>
</tr>
<tr>
<td>Hardness</td>
<td>55 Durometer</td>
</tr>
<tr>
<td>Tension</td>
<td>Approximately 120 pounds per inch.</td>
</tr>
<tr>
<td>Heated roll:</td>
<td>23 inches</td>
</tr>
<tr>
<td>Diameter</td>
<td>26.7 feet per second.</td>
</tr>
<tr>
<td>Surface</td>
<td>250° F.</td>
</tr>
</tbody>
</table>

During both of these runs, the steam in the steam chamber is shut off in order to avoid possible differences in percent moisture pickup. In other words, the two samples ("K" and "L") are plasticized with moisture during compacting only to the extent of their respective equilibrium moisture content after storage for two to three weeks at room temperature and atmospheric humidity as

From the data shown in Table C, in these directly comparable runs, it is clearly apparent that with the impregnated web (L), the degree of compaction is substantially higher than the degree of compaction attained with the unimpregnated web (K). It is also apparent that the compaction efficiency attained with the impregnated paper (L) is substantially greater (by 12.9%) than the compaction efficiency attained in processing the unimpregnated paper (K).

The improvement in efficiency of compaction attained with the impregnated web may be explained on the theory that the binder in the web exerts a "clutch action" upon the surface of the contractile rubber belt, increasing the frictional resistance of the web to any movement relative to the belt surface. Regardless, however, of the correctness of the suggested explanation, normally compaction of an impregnated paper under any given set of conditions in any given apparatus will produce a substantially higher degree of compaction and a higher efficiency of compaction than will be attained in processing an unimpregnated but otherwise identical paper in the same apparatus under substantially the same operating conditions.

The resulting unified paper according to this invention possesses remarkably improved delamination resistance for the cheap paper used and the relatively low amount of impregnant added and is extremely flexible and conformable, making it particularly suitable for a masking tape backing. The unified sheet of this example is coated on one side with the backsize composition of Example II and heated in the manner described in Example II to dry and cure the backsize. The resulting backsize layer weighs about 0.5 ounce per square yard. As in Example II, the opposite side of the sheet is coated with the following self-curing pressure-sensitive adhesive mass dissolved in toluene to about 33 percent solids to apply a pressure-sensitive adhesive layer weighing about 2.4 ounces per square yard:

### Self-Curing Mass

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade name</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyurethane</td>
<td>Polyurethane pale tape</td>
<td>87.5</td>
</tr>
<tr>
<td>Butadiene-styrene (50:50)</td>
<td>NR 100</td>
<td>12.5</td>
</tr>
<tr>
<td>Phenolic formaldehyde resin</td>
<td>85% formaldehyde resin</td>
<td>0.9</td>
</tr>
<tr>
<td>Aluminum hydride</td>
<td>Ammonol 46-88</td>
<td>10.0</td>
</tr>
<tr>
<td>2,4 di-tert. amylphenol</td>
<td>Aluminum hydride</td>
<td>10.0</td>
</tr>
<tr>
<td>2,4 di-tert. amylphenol</td>
<td>Stearoyl A</td>
<td>2.0</td>
</tr>
<tr>
<td>Toluene diisocyanate</td>
<td>TD1</td>
<td>2.5</td>
</tr>
</tbody>
</table>

The resulting pressure-sensitive adhesive sheet is slit
as described in Example II into rolls of tape wound in successive convolutions upon itself in the form of a roll with the pressure-sensitive layer facing inward. The tape rolls obtained do not delaminate when the tape is unwound rapidly from the roll and the tape exhibits extraordinary ability to conform to surfaces of various shapes. It also may be applied in sharp curves to flat surfaces without puckering at either edge of the tape. In other words the tape may be applied flat upon a flat surface in the form of a sharp curve. When used to mask a design of this type for spray painting, a sharply defined curved design is obtainable. The tape of this example is superior in this respect to competitive commercial tapes and yet is considerably less expensive due to the fact that it is made from a much cheaper starting web and much less impregnated, i.e., only about 15% by weight, is added to the backing.

EXAMPLE IX

A toweling grade “Kowtowi” web of the type described in Example VIII is impregnated with about 20% by weight of a high modulus, unplasticized, polyvinyl acetate resin in an aqueous dispersion containing about 15% solids. The impregnated web is compacted and cured as described in Examples I and VIII to provide a unified paper according to this invention with decidedly improved delamination resistance as indicated below:

<table>
<thead>
<tr>
<th>Paper:</th>
<th>Delamination resistance (ozs.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting web</td>
<td>8</td>
</tr>
<tr>
<td>Impregnated web</td>
<td>16.9</td>
</tr>
<tr>
<td>Compact impreg. web</td>
<td>26.4</td>
</tr>
</tbody>
</table>

The unified paper of this example is made into a pressure-sensitive masking tape in the manner described in Example VIII. The resulting tape is extremely flexible and conformable despite the high modulus impregnant employed and yet possesses improved firmness which facilitates handling and application of the tape to surfaces to be masked. There is no sign of delamination of the backing when the tape is unrolled and the tape is capable of conforming to surfaces of various shapes and being applied flat to define curved lines without puckering at the edges of the tape as described in Example VIII.

EXAMPLE X

The same “Kowtowi” starting web is impregnated as described in Example VIII using high modulus material of the following composition diluted to about 20 percent total solids by the addition of water.

<table>
<thead>
<tr>
<th>Material</th>
<th>Trade name</th>
<th>Parts by weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>Butadiene-styrene acrylate acid</td>
<td>Lotel 2722</td>
<td>48 3,102.0</td>
</tr>
<tr>
<td>Heat curing phenolic formaldehyde resin</td>
<td>Durex 14,796</td>
<td>65 1,063.0</td>
</tr>
<tr>
<td>Tetra sodium salt of ethylene diamine tetraacetic acid</td>
<td>Permalkote 80</td>
<td>33 21.2</td>
</tr>
<tr>
<td>Antioxidant</td>
<td>Aminec dispersion</td>
<td>52 2.6</td>
</tr>
<tr>
<td>Potassium polyacrylate</td>
<td>Acrysol G.S. diluted to</td>
<td>1.2 16.0</td>
</tr>
</tbody>
</table>

The impregnated web is dried as described in Example I so that about 20% solids by weight of the dry web are added. The web then is compacted and cured according to Examples I and VIII to provide a web having improved delamination resistance as indicated below:

<table>
<thead>
<tr>
<th>Paper:</th>
<th>Delamination resistance (ozs.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Impregnated</td>
<td>18.4</td>
</tr>
<tr>
<td>Compact impreg. web</td>
<td>30.4</td>
</tr>
</tbody>
</table>

The resulting paper is made into a masking tape as described in Examples VIII and IX which is extremely flexible and conformable and yet possesses good firmness for ease of application. This is another example of how a high modulus impregnant improves the firmness of the backing without seriously detracting from its conformability.

EXAMPLE XI

An artificial leather according to this invention is made from a relatively unhydrated flat bleached sulfate paper about 20 mils thick and having a basis weight of about 162 lbs., by first impregnating the paper with a water based acrylic latex to add about 21% impregnant solids by weight of the dry web and then compacting the impregnated web as described in Example I. The acrylic latex is one sold by Rohm and Haas of Philadelphia under the name Rhoplex B-15 and is believed to be essentially an ethyl acrylate polymer with a small percent of an acrylic acid modification. The impregnant composition comprises Rhoplex B-15 diluted to 25% solids and modified by the addition of 0.5% (solids on solids) of tetra-sodium ethylene diamine tetraacetic acid.

The following values are obtained for delamination resistance:

<table>
<thead>
<tr>
<th>Paper:</th>
<th>Delamination resistance (ozs.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting web</td>
<td>17</td>
</tr>
<tr>
<td>Control (compacted unimpregnated starting web)</td>
<td>36</td>
</tr>
<tr>
<td>Impregnated web</td>
<td>42</td>
</tr>
<tr>
<td>Compact impregnated web</td>
<td>67</td>
</tr>
</tbody>
</table>

The resulting artificial leather possesses unexpectedly high delamination resistance, i.e., 67 ozs. and when flexed retains its integrity and does not blister or split. It also is free from fiber picking, i.e., the fibers are unified and are not easily removed from the web, accidentally or otherwise. This paper also is unusually soft and pliable and therefore adapted to be substituted for leather in many applications.

EXAMPLE XII

The starting web of Example V is treated as described in Example V except that the acrylate impregnant of Example XI is substituted for the impregnant composition of Example I with results very similar to those of Example V. Values obtained for delamination resistance are given below:

<table>
<thead>
<tr>
<th>Paper:</th>
<th>Delamination resistance (ozs.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting web</td>
<td>16</td>
</tr>
<tr>
<td>Control (compacted unimpregnated starting web)</td>
<td>32</td>
</tr>
<tr>
<td>Impregnated web</td>
<td>24</td>
</tr>
<tr>
<td>Compact impregnated web</td>
<td>48</td>
</tr>
</tbody>
</table>

The resulting paper according to this invention is capable of many uses but is particularly suited for use as a tape backing because of its unexpectedly high delamination resistance and its extraordinary conformability.

EXAMPLE XIII

A flat moderately hydrated paper having a basis weight of about 32 lbs. and being about 4.6 mils thick and formed from bleached kraft softwood pulp is impregnated with an aqueous impregnant composition comprising about 30% of the following solids:

<table>
<thead>
<tr>
<th>Material</th>
<th>Parts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Buna N latex (33 acrylonitrile 67 butadiene)</td>
<td>88.4</td>
</tr>
<tr>
<td>Tetra sodium ethylene diamine tetraacetic acid</td>
<td>0.4</td>
</tr>
<tr>
<td>Red pigments</td>
<td>11.2</td>
</tr>
</tbody>
</table>

The percent impregnation, i.e., add-on, is about 50% by weight of the dry starting web. The impregnated web is compacted as described in connection with FIG. 4 by adjusting its moisture content to about 15% by weight of the web and then pasting the web through the com-
pacting unit to achieve a total degree of compaction of about 20% without substantially increasing the thickness of the web. The resulting paper according to this invention has improved flexibility, tear strength, and delamination resistance.

The unified paper of this example then is made into a sandpaper. First, one of its sides is coated with a binder coat comprising a polyvinyl chloride resin plastidized with a copolymer of butadiene and acrylonitrile, and then a binder coat based upon a phenol-formaldehyde resin is applied to the barrier layer. The binder coat is several times thicker than the barrier coat and sand granules are applied to the paper by partially embedding them in the wet binder coat. After drying the binder coat, a conventional sand size next is applied over the binder layer to provide a sandpaper according to this invention which possesses superior flexibility, tear strength and crack resistance.

**EXAMPLE XIV**

A rope paper of cylinder machine manufacture of 18 lbs. raw weight (24 x 36 x 480) is compressed to 65% of its original length by treatment in a laboratory adaptation of the equipment described hereinabove and illustrated in the drawings.

An untreated sample of the paper showed the following characteristics:

- M.D. tensile strength 24 lbs./in.
- Caliper .002 inch.
- Gurley density 45 sec. (2 ply 400 cc.) 5 oz. cylinder 1/4’’ opening.

The above treated samples show M.D. tensile strength of 8 lbs./in.

- Caliper .0045 inch.
- Gurley density 25 sec. (2 ply 400 cc.) 5 oz. cylinder 1/4’’ opening.

Samples of treated and untreated paper are impregnated in an aqueous colloidal dispersion of 33% total solids comprising as the dispersed phase:

- 66% by weight of a copolymer of butadiene and acrylonitrile in the ratio of 75 parts butadiene to 25 parts of acrylonitrile emulsion polymerized in the presence of a fatty acid soap to a Mooney of 40.
- 33.5% by weight of a copolymer of butadiene and styrene emulsion polymerized to a Mooney of 35.
- 0.5% of polymer solids of an antioxidant for the rubber.

Tests on the treated paper and on a control saturated to 100% by weight of solids of the above latex mixture show:

**Control**

- Texture, 24.7#/in. 28.6#/in.
- Delamination resistance, 100+ oz. 60 oz.

**EXAMPLE XV**

A flat kraft paper of 38 lbs. basis weight (24 x 36 x 480) of the following characteristics:

- M.D. tensile 21 lbs./in.
- Gurley density 100 sec. 100

cannot be impregnated in the untreated state. After treatment by longitudinal compression to the extent of reduction in length of 10%, the sheet can be saturated.

Physical properties of the treated sheet are:

- M.D. tensile 10 lbs./in.
- Gurley density 52 sec.

Physical properties of the treated sheets saturated to the extent of 80% of the raw weight with the latex mixture described in Example I are:

- M.D. tensile 22.5 lbs./in.
- M.D. elongation 14.8% elongation.
- Delamination resistance 35 oz.

**EXAMPLE XVI**

A rope paper of cylinder machine manufacture of 30 lbs. basis weight (24 x 36 x 480) is compressed in the cross machine direction in a laboratory adaption of the equipment shown in the drawings. One sample of the sheet is passed through the compression machine once and another sample twice.

Both samples and a control are impregnated in an aqueous colloidal dispersion of 35% total solids comprising as the dispersed phase:

- 75 parts by weight of a copolymer of butadiene and acrylonitrile in the ratio of 75 parts butadiene to 25 parts of acrylonitrile emulsion polymerized in the presence of a fatty acid soap to a Mooney of 40.
- 25 parts by weight of a copolymer of butadiene and styrene emulsion polymerized to a Mooney of 35.

The impregnated samples then are dried and heated at 380° F. for 1 minute in a circulating-air oven. Tests on the compressed samples and the control give the following results:

**Sample:**

- Delamination resistance
  - Control 56 ounces
  - 1 pass compression sample 60 ounces
  - 2 pass compression sample 60 ounces 133

Cellulosic fibers of various sizes and shapes and of various conditions of hydration may be used in forming papers according to this invention. Typically, the starting web may comprise a major proportion of softwood kraft pulp preferably bleached or semi-bleached and slightly to moderately hydrated. Other cellulosic fibers such as rope fibers may be used as the major constituent and other fibers including hardwood fibers and even certain non-cellulosic fibers may be used as the minor constituent of the starting web in papers according to this invention. As mentioned hereinbefore, the starting web may be formed from fibers which are substantially straight and possess only natural curl or it may include a substantial proportion of fibers to which a substantial amount of curl or crimp has been imparted prior to web formation.

A variety of materials may be used as the impregnant, or a defining agent, for bonding the fibers in unified papers according to this invention. The term "unified papers" means primarily papers possessing increased resistance to splitting but is intended to also include increased resistance to fiber removal by spot picking or surface abrasion. Similarly, a "unifying amount" of the impregnant means that amount which prevents the paper from delaminating under normal conditions of use. These unifying materials may be classified generally as nonvolatile extendible polymers, extensible polymeric fiber bonding agents, elastomeric fiber bonding agents, or the like, and may be applied from water or from solvent based systems such as those based on volatile organic solvents, depending upon the material. They also may be classified as high and low modulus impregnants, both of which may be used in making papers according to this invention, as described hereinbefore.

The high modulus impregnants or polymers are in fact preferred at low levels of impregnation, i.e., about 10–30% by weight of the dry web. These high modulus materials, in the transition range of viscoelastic behavior, as described hereinbefore, include polystyrene, polystyrene copolymers, high styrene copolymers, such as high styrene (60–90% styrene) butadiene-styrene copolymers, polyethylene, polyvinyl acetate, polyvinyl chloride, polyvinylidene chloride, polyvinyl acetal, polyeurathanes, polyesters, and the like.

Suitable high modulus impregnants may be compounded by adding large amounts of modifying resins in conventional low modulus elastomer systems. For instance,
Large amounts of heat curing phenolic resins may be used with chloroprene, isoprene, butadiene-styrene, butadiene-acrylonitrile or alkyl acrylate ester polymers and copolymers in such systems. Polysiocyanates and blocked polyisocyanates also may be used in the impregnant to increase the strength and modulus of the impregnant and to improve the bond between the polymer and the paper fibers.

Conventional low modulus extensible polymers also are suitable as impregnants in this invention. These include natural rubbers; diene polymers and copolymers such as: butadiene, isoprene and chloroprene polymers; copolymers of these diene monomers with styrene, acrylonitrile, acrylic, or methacrylic acid esters; isobutylene polymers and copolymers; alkyl acrylate ester polymers and copolymers; polyvinyl ethers; glue-glycerine; and the like.

As mentioned hereinbefore the order of impregnation and compaction may be reversed although it is preferred to add the impregnant before compaction, and in certain cases it may be advantageous to perform impregnation and compaction in more than two steps. Another modification of the invention involves deposition of the unifying agent on the fibers prior to sheet formation. This prior treatment of the fibers with the bonding agent may be carried out by means of any of the known interpenetrating impregnation techniques and is most advantageously used when the compression mechanism is set up in connection with a paper machine. However, the principle may be modified by drying and rewetting the beater impregnated paper; or the paper, of suitable moisture content, may be stored in a moist condition and compressed after a convenient time interval. The fiber bonding materials applicable in this modification are limited to those which can be deposited by beater impregnation. The quantities of fiber bonding agents relative to fiber are limited naturally to the highest quantity which can be deposited onto the fibers. Usually somewhat less than the maximum quantity which can be deposited will make paper machine operation easier and result in an equal or superior final product. The lower limit may be determined only by evaluation of the final product for the use intended.

As indicated herefore, the unifed papers of this invention are particularly useful as backings for pressure-sensitive adhesive tapes. The paper backing may be coated in a suitable manner with a suitable adhesive mass to form a new and improved adhesive tape. The adhesive mass used may be any of the conventionally used tacky solvent-adhesive, or normally tacky and pressure sensitive adhesive masses that are sold known in the prior art. Most of the commonly used pressure-sensitive adhesives are based on relatively non-polar elastomers such as natural rubber, polysisoprene and the vulcanizable elastomeric copolymers and interpolymers of at least 40 percent butadiene and at least 10 percent styrene. Such an adhesive mass may be especially compounded, copolymerized or interpolymerized to have the desired degree of adhesiveness in absence of additional tackifier. Or, alternatively, any of the conventional tackifiers may be used such as rosin, hydrogenated rosin, dehydrogenated rosin, the glycols and glycerides of any of these resinous materials, polyterpenes, coumarone indene resins, poly-styrene, oil-soluble aldehyde resins, or any other desired tackifier in any of the proportions that are well known in the art. The adhesive mass may be free of a filler or may contain inert filler such as zinc oxide, magnesium carbonate, calcium carbonate, lead oxide, clay, titanium dioxide, aluminum, hydrated alumina, silica or any of the other conventional fillers for normally tacky and pressure-sensitive adhesives. Other ingredients such as anti-oxidants or heat stabilizers, dyes or pigments may be present or absent depending upon the particular desired use for the adhesive sheet. Typical normally tacky and pressure-sensitive adhesive masses are disclosed in Johnson & Johnson's British Patent No. 611,211 issued October 1948; in Nelson's United States Patent No. 2,415,901 issued February 18, 1947; in Priepke's United States Patent No. 2,405,926 issued August 13, 1945; in Buckley's United States Patent No. 2,397,774 issued April 2, 1946; and in many other patents. Exemplary of suitable normally non-tacky adhesives are conventional water-activatable adhesive, organic solvent-activatable resins such as those based upon vinyl chloride polymers, etc.

If desired, a priming coating may be interposed between the impregnated paper backing and the adhesive mass in order to secure improved adhesion of these two laminates. Any suitable priming coatings may be employed as, for example, those disclosed in the United States patent to Morria, No. 2,424,996. For ease in unrolling the tape, it also is desirable to provide upon the surface of the backing, opposite that to which the adhesive mass is applied, a coating which decreases the adhesion of the adhesive mass to the face surface of the pressure-sensitive adhesive tape. Any suitable conventional release coating for such products may be employed as, for example, a lacquer, a surface-active material of the type disclosed in the United States patent to Collins, No. 2,913,355, etc.

When the paper backing for pressure-sensitive adhesive tape according to this invention is formed by compaction the paper horizontally by at least about 5% to about 40% of its length, the tape resulting from processing as described above normally will be extensible to the extent of at least about 5% to about 40% in length when stretched lengthwise.

An example of such a tape according to this invention is shown in FIG. 3 wherein a coating 2 of a normally tacky and pressure-sensitive adhesive is applied to one side of a unified sheet produced in accordance with this invention, which sheet forms the backing of the tape.

Having now described the invention in specific detail and exemplified the manner in which it may be carried into practice, it will be readily apparent to those skilled in the art innumerable variations, applications, modification, and extensions of the basic principles involved may be made without departing from its spirit or scope.

What is claimed is:

1. A pressure-sensitive adhesive masking tape comprising a unified paper backing coated on one surface thereof with a pressure-sensitive adhesive, said tape being formed in successive convolutions upon itself to form a roll from which it is rapidly unwindable without delamination of said paper backing, and when so unwound, is extensible to the extent of at least about 5% in length when stretched lengthwise; said unified paper backing being composed of distorted, longitudinally crowded cellulose fibers, bonded together by about 10-30% by weight of the dry fibers of a high modulus extendible polymeric unifying material distributed within the body of the backing to increase the firmness and delamination resistance of the backing, said unifying material being in the transition range of viscoelastic behavior, the fibers of said backing being distorted and longitudinally crowded by subjecting a starting web having an elongated web body composed of cellulose fibers and having two opposed web faces, while heated to a temperature sufficient to generate moisture vapor, simultaneously to (a) sufficient confining pressure, acting perpendicularly with respect to said web faces to prevent substantial wrinkling of the web body, and (b) sufficient compressing force, acting longitudinally with respect to the length of said web (i) to cause the web fiber to be longitudinally distorted and forced closer together in changed orientation throughout within the web body and (ii) to cause the web body thereby to be act in length by at least about 5%; whereby an inexpensive tape is provided characterized by (a) high resistance to delamination, (b) high conformability, and (c) good firmness.

2. A unified, compacted paper sheet adapted to be used as a backing for adhesive tape, abrasive sheet ma-
terial, artificial leather and the like; said paper sheet comprising (a) cellulosic papermaking fibers that are compressively distorted, and crowded together within the body of said sheet and (b) an extendible, polymeric fiber-bonding agent, distributed substantially throughout the body of said sheet in unifying amount and in fiber-bonding relationship with said cellulosic fibers to bond said fibers in their distorted and crowded state; said unified, compacted paper sheet being formed by compaction of a paper starting web having a sheet-like web body composed essentially of cellulosic papermaking fibers, and a median plane lying generally between its web faces, by carrying out the following operations upon said starting web, to wit:

(1) incorporating into said web a polymeric fiber-bonding agent in a proportion which is unifying after compaction, thereafter
(2) subjecting said polymer-containing starting web, while plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, simultaneously to:

(a) sufficient compacting force, acting in a direction generally parallel to said median plane, substantially to contract the area of said web body and to distort and crowd said fibers closer together in changed orientation with respect to each other, within said web body between said web faces; and
(b) sufficient confining force, acting in a direction generally perpendicular to said median plane, to prevent substantial crinkling of said web body during such compaction; and

(3) drying said compacted web to bond the distorted fibers in their changed orientation.

3. A unified compacted paper according to claim 2, wherein the amount of the extendible polymeric fiber-bonding agent present is at least about 10% by weight of the dry web.

4. A unified compacted paper according to claim 3, wherein the amount of the extendible polymeric fiber-bonding agent present is not above about 50% by weight of the dry web and said fiber-bonding agent is in the transition range of viscoelastic behavior.

5. A unified, compacted paper sheet according to claim 2, wherein said starting web is creped.

6. A unified, compacted paper sheet according to claim 2, wherein said starting web is uncreped.

7. A relatively dense, unified, compacted paper sheet adapted to be used as a backing for adhesive tape, abrasive sheet material, artificial leather and the like; said paper sheet comprising (a) cellulosic papermaking fibers that are compressively distorted, crowded and pushed together within the body of said sheet and (b) an extendible, polymeric fiber-bonding agent, distributed within the body of said sheet in unifying amount and in fiber-bonding relationship with said cellulosic fibers to bond said fibers in their distorted and crowded state; said unified, compacted paper sheet being formed by a paper web comprising of cellulosic papermaking fibers and having a web body between two opposed, substantially parallel web faces, by carrying out the following operations upon said paper web, to wit:

(1) incorporating into said web a unifying proportion of said polymeric fiber-bonding agent; thereafter (2) simultaneously (a) applying force, acting perpendicularly with respect to said web faces, to prevent substantial distortion of said paper containing web body and to maintain said web faces substantially parallel to each other, and (b) compacting the web in a direction perpendicular to said web faces while said paper containing web body is plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, thereby (i) to distort and crowd the web fibers together horizontally in changed orientation within the web body and (ii) to contract the area of the web body by at least 5% to about 40%; and (3) drying said compacted web to bond the distorted fibers in their changed orientation; said unified compacted paper sheet being characterized by (a) high resistance to delamination and (b) substantial extendibility when stretched in a direction parallel to the direction of compaction.

8. A relatively dense, unified, compacted paper sheet comprising (a) cellulosic papermaking fibers that are compressively distorted, crowded, and pushed together within the body of said sheet and (b) an extendible, polymeric fiber-bonding agent, distributed within the body of said sheet in unifying amount and in fiber-bonding relationship with said cellulosic fibers to bond said fibers in their distorted and crowded state; said unified, compacted paper web being formed from a paper web composed of cellulosic papermaking fibers and having a web body between two opposed, substantially parallel web faces, by carrying out the following operations upon said paper web, to wit:

(1) incorporating into said web a unifying proportion of said polymeric fiber-bonding agent; thereafter (2) simultaneously (a) applying force, acting perpendicularly with respect to said web faces, to prevent substantial crinkling of said polymeric fiber-bonding agent; and (3) maintaining said web faces substantially parallel to each other, and (b) compacting the web in a direction parallel to said web faces while said polymeric-containing web is plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, thereby (i) to distort and crowd the web fibers together horizontally in changed orientation within the web body and (ii) to contract the area of the web by at least 5% to about 40%; and (3) drying said compacted web to bond the distorted fibers in their changed orientation; said unified compacted paper sheet being characterized by (a) high resistance to delamination and (b) substantial extendibility when stretched in a direction parallel to the direction of compaction.
In the web body, to be longitudinally distorted and forced closer together in changed orientation lengthwise within the web body, and (ii) thereby to cause the web body to contract in length by at least from about 5% to about 40%; and (f) drying the contracted web, thereby to retain the distorted fibers extensively bonded together in their changed orientation; whereby to obtain a roll of pressure-sensitive tape that is rapidly unwindable from the roll without delamination of said unified backing; and when so unwound, is extensible to the extent of at least about 5% to about 40% in length when stretched lengthwise.

10. In the manufacture of a unified, extensible cellulosic fibrous paper sheet adapted to be used as a backing for adhesive tape, abrasive sheet material, artificial leather and the like; that improvement which comprises: (1) incorporating in a cellulosic fibrous paper web a unifying proportion of an extensible polymer in fiber-bonding relationship with the cellulosic fibers thereof; thereafter (2) simultaneously (a) applying force, acting perpendicularly with respect to the principal surfaces of the web, to prevent crinkling of the body of the web, and maintain said principal web surfaces substantially parallel to each other and (b) compacting the web, horizontally with respect to the principal surfaces of the web, while said polymer-containing web is plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, whereby (i) to distort and crowd the web fibers together horizontally in changed orientation within the body of the web and (ii) to contract the area of the web by at least about 5% to about 40%; and (3) drying the compacted web containing said extensible binder to internally bond the distorted fibers in their changed orientation; whereby to obtain a unified, compacted, internally bonded, cellulosic fibrous web characterized by (a) high resistance to delamination and (b) substantial extensibility when stretched in a direction parallel to its direction of compaction.

11. The improvement of claim 10 wherein said extensible polymer is incorporated in a cellulosic fibrous web by beating impregnation during web formation thereof.

12. The improvement of claim 10 wherein said extensible polymer is incorporated in a cellulosic fibrous web by impregnating a preformed cellulosic fibrous web with said polymer dissolved in a volatile organic solvent.

13. An improvement of claim 10, wherein said web is plasticized during said compacting step by a moisture content not substantially greater than said web's equilibrium moisture content when stored at a room temperature at about 50% relative humidity.

14. In the manufacture of a unified, extensible cellulosic fibrous paper sheet adapted to be used as a backing for adhesive tape, abrasive sheet material, artificial leather and the like; that improvement which comprises: (1) incorporating in a paper web of papermaking cellulosic fibers a unifying proportion of an extensible polymer in fiber-bonding relationship with said cellulosic fibers by impregnation of said paper web with an aqueous dispersion of said polymer; thereafter (2) simultaneously (a) applying force, acting perpendicularly with respect to the principal surfaces of said paper web, to prevent substantial crinkling of the body of the web and to maintain said principal surfaces substantially parallel to each other, and (b) compacting said paper web, in a direction parallel to said principal surfaces, while said polymer-containing paper web is plasticized with moisture and heated to a temperature sufficient to generate moisture vapor whereby (i) to distort and crowd said cellulosic fibers horizontally together in changed orientation within the body of the paper web and (ii) to contract the area of the paper web by at least about 5% to about 40%; and (3) drying the polymer-containing compacted paper web to internally bond the distorted fibers in their changed orientation; whereby to obtain a unified compacted paper web characterized by (a) high resistance to delamination and (b) substantial extensibility when stretched in a direction parallel to its direction of compaction.

15. A pressure-sensitive adhesive tape comprising a unified paper backing coated on one surface thereof with a normally tacky and pressure-sensitive adhesive, said tape being wound in successive convolutions upon itself to form a roll from which it is rapidly unwindable without delamination of said backing; said backing being composed of (a) paper-making cellulosic fibers that are compressively distorted and crowded together within the body of the backing, and (b) an extensible, polymeric, fiber-bonding agent, distributed substantially throughout the body of such backing in unifying amount and in fiber-bonding relationship with said fibers to bond said fibers together in their distorted and crowded state; said backing being formed by carrying out the following operations, to wit:

(1) providing a starting web comprising an open, porous paper web composed essentially of paper-making cellulosic fibers, said starting web having a sheet-like web body with a median plane lying generally between its web faces;

(2) subjecting said starting web, while plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, simultaneously to:

(a) sufficient compacting force, acting in a direction generally perpendicular to said median plane, substantially to contract the area of said web body and to distort and crowd said fibers closer together in changed orientation with respect to each other, within said web body between said faces; and

(b) sufficient confining force, acting in a direction generally perpendicular to said median plane, to prevent substantial crinkling of said web body during such compaction;

thereby to produce a compacted intermediate web that is

(i) nonuniform, (ii) less absorbent than said starting web and (iii) structurally stable in the presence of moisture; and

(3) thereafter unifying said compacted intermediate web by impregnation with a fiber-bonding agent comprising an extensible polymer to distribute a unifying proportion of said fiber-bonding agent substantially throughout the body of said intermediate web, in fiber-bonding relationship with said distorted fibers and thereby to produce a compacted and unified web having a delamination resistance that is substantially greater than the delamination resistance of an uncompacted but otherwise identical impregnated web.

16. A pressure-sensitive adhesive tape according to claim 15, wherein the amount of the extensible polymeric fiber-bonding agent present is about 10-50% by weight of the dry web and said fiber-bonding agent is in the transition range of viscoelastic behavior.

17. A pressure-sensitive adhesive tape comprising a unified paper backing coated on one surface thereof with a normally tacky and pressure-sensitive adhesive, said tape being wound in successive convolutions upon itself to form a roll from which it is rapidly unwindable without delamination of said backing; said backing being composed of (a) paper-making cellulosic fibers that are compressively distorted and crowded together within the body of the backing, and (b) an extensible, polymeric, fiber-bonding agent, distributed substantially throughout the body of such backing in unifying amount and in fiber-bonding relationship with said fibers to bond said fibers together in their distorted and crowded state; said tape being formed by carrying out the following operations, to wit:

(1) providing a starting web comprising an open, porous paper web composed essentially of paper-making cellulosic fibers, said starting web having a sheet-
like web body with a median plane lying generally between its web faces;
(2) subjecting said starting web, while plasticized with moisture and heated to a temperature sufficient to generate moisture vapor, simultaneously to:

(a) sufficient compacting force, acting in a direction generally parallel to said median plane, substantially to contract the area of said web body and to distort and crowd said fibers closer together in changed orientation with respect to each other, within said web body between said faces; and

(b) sufficient confining force, acting in a direction generally perpendicular to said median plane, to prevent substantial wrinkling of said web body during such compaction;

thereby to produce a compacted intermediate web that is (i) nonunified, (ii) less absorbent than said starting web and (iii) structurally stable in the presence of moisture;

(3) thereafter unifying said compacted intermediate web by impregnation with a fiber-bonding agent comprising an extensible polymer to distribute a unifying proportion of said fiber-bonding agent substantially throughout the body of said intermediate web, in fiber-bonding relationship with said distorted fibers and thereby to produce a compacted and unified web having a delamination resistance that is substantially greater than the delamination resistance of an uncompacted but otherwise identical impregnated web;

(4) coating one surface of said compacted, unified paper web with a pressure-sensitive adhesive mass; and

(5) winding said coated web in successive convolutions

upon itself to form a roll of pressure-sensitive adhesive tape that is rapidly unwindable from said roll without delamination of said compacted, unified and coated web.

19. The product of claim 18 wherein said web, during the application of said compacting force, is contracted in area by at least 5% to about 40%.

20. The product of claim 19 wherein said unifying operation is carried out by impregnating said compacted intermediate with said fiber-bonding agent dispersed in a volatile organic solvent and thereafter evaporating said volatile solvent.

21. The product of claim 19 wherein said unifying operation is carried out by impregnating said compacted intermediate with said fiber-bonding agent dispersed in an aqueous vehicle and thereafter evaporating said aqueous vehicle.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,055,496

September 25, 1962

Isaac R. Dunlap

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 2, line 10, strike out "to"; column 3, line 73, for "exhaustive" read -- extensive --; column 9, line 52, for "like" read -- life --; column 10, line 62, after "have" insert -- a --; column 29, line 22, before "crinkling" insert -- substantial --.

Signed and sealed this 5th day of February 1963.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents