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54 **Kraft liner board and method of producing Kraft liner board from bleached or unbleached Kraft pulp, TMP pulp, SCMP or sulfite pulp.**

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57 The invention relates to a method of improving the wet strength of paper products produced from bleached or unbleached kraft pulp, thermomechanical pulp, SCMP or sulfite pulp. The method comprises the steps of subjecting paper to high pressure densification during its production and heating the paper so as to raise its internal temperature to at least 216°C for a period of time sufficient to increase the wet strength thereof.

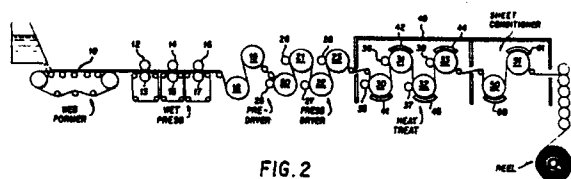


FIG. 2

"IMPROVING KRAFT LINERBOARD FROM UNBLEACHED KRAFT PULP, BLEACHED KRAFT PAPERBOARD, TMP PAPERBOARD, AND PAPERBOARD FROM SCMP AND SULFITE PULP".

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION:

This invention relates to the art of papermaking, particularly to treating a paper products with pressure and heat to improve its wet strength while preserving its folding endurance. This invention, more specifically, relates to Kraft paper products, bleached or unbleached paper products produced from SCMP and sulfite pulps, TMP paperboard. TMP paperboard means thermomechanical pulp paperboard. The term "SCMP" process means semichemical mechanical pulping process.

Description of the Prior Art:

The kraft process is a method of preparation of an aqueous slurry of fibers by treatment of a suitable renewable raw material. In most pulping process, a considerable portion of the natural lignin in wood, grass or other vegetative matter is rendered soluble by chemical reaction with one or more nucleophilic reagents. In the kraft process, the nucleophilic reagents are sulfide and hydroxide ions, which are used under highly alkaline conditions. Variations of the kraft process include the earlier practiced soda process, using hydroxyl ions derived from metals in Group IA of the periodic table, namely lithium, sodium, potassium, rubidium and cesium. A second variation involves the use of anthraquinone (AQ) or substituted anthraquinones as additional nucleophiles. Anthraquinone can be used in the soda process, in which case the process is known as the soda-AQ process, or in the kraft process which is then known as the kraft-AQ process. Such variations in the kraft process are well known in the industry and pulps prepared by any of these variations can be used in practicing the present invention.

If desired, the soda-AQ, kraft and kraft AQ pulps can be rendered white by application of suitable bleaching agents. Such agents are usually electrophilic in nature and may include chlorine, chlorine dioxide, sodium hypochlorite, hydrogen peroxide, sodium chlorite, oxygen and ozone. Use is often in sequential stages and a suitable nucleophilic agent, customarily hydroxyl ion, may be used in intermediate stages. "Kraft paper" is paper made from pulp produced by the kraft process. Bleached kraft paper, because of its low lignin content, has low wet strength; hence it is

desirable to develop this quality of bleached kraft products.

Linerboard is a medium-weight paper product used as the facing material in corrugated carton construction. "Kraft linerboard" is linerboard made from pulp produced by the kraft process.

The thermomechanical pulping process is a method of producing an aqueous slurry of fibers by mechanical treatment of a suitable renewable raw material. In most pulping processes, a considerable portion of the natural lignin in wood, grass or other vegetative matter is rendered soluble by chemical reaction with one or more nucleophilic reagents. Such nucleophilic reagents are not used in thermomechanical pulp production. Instead, fibers are liberated from a thermally softened source material, usually wood chips, by passage through a suitable shredder or disc refiner operating under ambient steam pressure at a temperature of 120-150°C or more. Such fibers are usually brittle and sheets prepared from them are brittle and have little or no wet strength.

As used herein, "TMP pulp" is pulp produced as described above, by a thermomechanical process. Despite the fiber brittleness, TMP pulp has the advantage of avoiding the odor, disposal problems, and other drawbacks of chemical pulp production. "TMP paperboard" is a medium-weight paper product made from TMP pulp. It is notoriously weak when wet, and inflexible, compared with products identically made from more flexible pulps such as kraft pulps. Hence, a process for improving both the wet strength and flexibility of TMP paper products is desirable.

In the art of making kraft paper products, TMP paperboard and SCMP paperboard, it is conventional to subject felted fibers to wet pressing to unite the fibers into a coherent sheet. Pressure is typically applied to a continuous running web of paper by a series of nip rolls which, by compressing the sheet, both increases its volumetric density and reduce its water content. The accompanying Fig. 1 shows in simplified diagrammatic form a typical papermaking machine, including a web former and three representative pairs of wet press rolls. Also shown are drying rolls whose purpose is to dry the paper to a desired final moisture content, and a calender stack to produce a smooth finish. At least some of the rolls are ordinarily heated to hasten drying. (The drawing is simplified -there are many more drying rolls in actual practice.)

The semichemical mechanical pulping process is a method of production of an aqueous slurry of fibers by treatment of a suitable renewable raw

material. In most pulping processes, a considerable portion of the natural lignin in wood, grass or other vegetative matter is rendered soluble by chemical reaction with one or more nucleophilic reagents. Minimization of the lignin portion solubilized and removed whilst so altering the lignin as to permit recovery of fibers by the mechanical action of a disk or other refiner or shredder in a condition of little damage is the goal of the SCMP process. It is also the goal of certain related processes known as the chemimechanical process (CMP), the chemithermomechanical process (CTMP) and the neutral sulfite semichemical (NSSC) processes. Such pulps are normally considered to be more brittle and of inferior strength when compared to lower lignin pulps produced by the kraft, sulfite, kraft-anthraquinone (AQ), soda-AQ or alkaline sulfite AQ processes. However, properties are adequate for many end-uses, including corrugated medium and even as a linerboard component.

In the sulfite process, sulfite or bisulfite ion is the nucleophilic agent. The sulfite or bisulfite ions cause the lignin molecules to break into small fragments. During this chemical reaction, the sulfite or bisulfite ions become chemically bonded to the lignin fragments thereby providing water solubility. A variation of the sulfite process involves the use of anthraquinone (AQ) or substituted anthraquinones as a second nucleophile. AQ is reduced in situ during the earliest stages of the cook to anthrahydroquinone (AHQ). As AQ is insoluble and only the salt form of AHQ is soluble, alkali presence is necessary for solution formation and uniform penetration of AQ into the wood chip, grass stem or any other fiber-containing vegetative matter. Such a cooking process is known as an alkaline sulfite-AQ process. Both the sulfite process and the alkaline sulfite-AQ variation of the sulfite process are well known to the industry and pulps thus prepared can be used to give the benefit of our invention.

There is currently considerable interest in treatments involving heat and pressure, or heat alone, during or after the production process, to improve various qualities of paper products. Quantifiable paper qualities include dry tensile strength, wet tensile strength, reverse folding endurance, compressive strength and stiffness, among others. Which qualities should desirably be enhanced depends upon the intended application of the products. For paper and TMP paperboard to be used in humid or wet environments, two qualities of particular interest are wet strength and folding endurance, both of which can be measured by well-known standard tests. As used herein, then, "wet strength" means wet tensile strength as measured by American Society for Testing and Materials - (ASTM) Standard D829-48. "Folding endurance" is

defined as the number of times a board can be folded in two directions without breaking, under conditions specified in Standard D2176-69. "Basic weight" is the weight per unit area of the dried end product.

For linerboard to be used in manufacturing corrugated cartons for use in humid or wet environments, three qualities of particular interest are wet strength, folding endurance and high humidity compression strength. "Compression strength" is edgewise linear compression strength as measured by a standard STFI (Swedish Forest Research Institute) Tester.

Prior workers in this field have recognized that high-temperature treatment of linerboard can improve its wet strength. See, for example E. Back, "Wet stiffness by heat treatment of the running web", Pulp & Paper Canada, vol. 77, No. 12, pp. 97-106 (Dec. 1976). This increase has been attributed to the development and cross-linking of naturally occurring polysaccharides and other polymers, which phenomenon may be sufficient to preserve product wet strength even where conventional synthetic formaldehyde resins or other binders are entirely omitted.

It is important to note that wet strength improvement by heat curing has previously been thought attainable only at the price of increased brittleness (i.e., reduced folding endurance). Therefore, most prior high-temperature treatments have been performed on particle board, wallboard, and other products not to be subjected to flexure. The known processes, if applied to bleached kraft paper, TMP paperboard and SCMP paperboard, would produce a brittle product. Embrittled paper and paperboard are not acceptable for many applications involving subsequent deformation such as the converting operation on a corrugating machine to make corrugated boxes out of linerboard, and therefore heat treatment alone, to develop wet strength of linerboard, has not gained widespread acceptance, and is not viable for TMP paperboard. As Dr. Back has pointed out in the article cited above, "The heat treatment conditions must be selected to balance the desirable increase in wet stiffness against the simultaneous embrittlement in dry climates." Significantly, in U. S. Patent 3,875,680, Dr. Back has disclosed a process for heat treating already manufactured corrugated board to set previously placed resins, the specific purpose being to avoid running embrittled material through a corrugator.

It is plain that added wet strength and improved folding endurance were previously thought incompatible results.

It is therefore an object of the invention to produce paper products and TMP paperboard and linerboard having both greatly improved wet

strength and good folding endurance. Another goal is to achieve that objective without resorting to synthetic resins or other added binders and wet strength agents.

With a view of the foregoing, a process has been developed which dramatically and unexpectedly increases not only the wet strength of paper products, TMP Paperboard, SCMP paperboard and linerboard, but also preserves its folding endurance. In its broadest sense, the invention comprises steps of 1) subjecting paper or linerboard, produced from bleached or unbleached kraft pulp or paperboard produced from thermomechanically produced pulp or SCMP paper pulp, to high pressure densification, and 2) heating the product to an internal temperature of at least 420°F (216°C) for a period of time sufficient to increase the wet strength of the product.

This method produces a product having folding endurance greatly exceeding that of similar products whose wet strength has been increased by heat alone. This is clearly shown by our tests exemplified below.

While the tests set out in the Examples have carried out the invention in a static press, it is preferred that the heat and pressure be applied to continuously running paper and board by hot pressure rolls, inasmuch as much higher production rates can be attained.

In the case of linerboard from unbleached kraft, we prefer to raise the internal temperature of the board to at least 550°F (289°C), as greater wet strength is then achieved. This may be because at higher temperatures, shorter step duration is necessary to develop bonding, and there is consequently less time for fiber degradation to occur. Also, shorter duration enables one to achieve higher production speeds.

In the case of other paper products from kraft pulp, and TMP boards, we prefer to raise the internal temperature of the paper to at least 465°F (240°C), as greater wet strength is then achieved. In the case of SCMP paperboard, it is preferred to raise the internal temperature of the board to at least 450°F (232°C).

It should be noted that the heating rate, and thus the required heating duration at a particular temperature, depends on method of heat transfer chosen. Furthermore, it is desirable to raise the web temperature as rapidly as possible to the chosen treating temperature. Improved heating rates can be achieved by using high roll temperatures and/or by applying high nip forces to the press roll against the sheet on the hot rolls. That high pressure dramatically improves heat transfer rates has previously been disclosed. One worker has attributed this to the prevention of vapor formation at the web-roll interface.

While the invention may be practiced over a range of temperatures, pressures and durations, these factors are interrelated. For example, the use of higher temperatures requires a heating step of shorter duration, and vice-versa. At 550°F, in the case of linerboard, a duration of 2 seconds has been found sufficient to obtain the desired improvements, while at 420°F, considerably longer time is required. At 465°F, a duration of 60 seconds has been found sufficient to obtain the desired improvements, while at 420°F, considerably longer time is required. In the case of SCMP board, at 450°F, a duration of 5 seconds has been found sufficient to obtain substantial improvement.

It is presently preferred that, for safety reasons, the roll temperature be not greater than the web ignition temperature (572°F, 300°C); however, even higher roll temperatures may be used if suitable precautions, such as the provision of an inert atmosphere, or rapid removal of paper from the hot environment, are taken.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows, in greatly simplified diagrammatic form, a conventional apparatus for producing linerboard.

Figure 2 shows, in like diagrammatic form, an apparatus for practicing the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Figure 2 illustrates a preferred apparatus for carrying out the inventive process, although it should be understood that other devices, such as platen presses, can be used and in fact the data below was obtained from platen press tests. In the machine depicted, bleached or unbleached kraft paper fibers or unbleached TMP pulp fibers or SCMP or sulfite pulp fibers in aqueous suspension are deposited on a web former screen 10, producing a wet mat or fibers. The mat is then passed through a series of wet press nip rolls 12, 13, 14, 15, 16 and 17 which develop a consolidated web. Suitable wet presses known today include long nip presses and shoe-type presses capable of developing high unit press pressures on the wet fiber web. This step is known as "high pressure wet pressing". The web is then passed over pre-drying rolls 18, 19 to remove water from the wet web. Once the moisture content of the web has been reduced to less than 70% by weight, steps of the high pressure densification and high temperature treatment are applied according to the invention.

To densify the web, a series of drying rolls 20, 21, 22, 23 are provided with respective pressure rollers 25, 26, 27, 28 which are loaded sufficiently to produce a web density of at least 700 kg/m³. We define this step as "press drying" or "high pressure densification". In the preferred embodiment, the high pressure densification step of the invention is carried out both at normal drying temperatures - (substantially below 400°F) in the press drying section, and also in the high temperature heat treatment section described below. It should be understood, however, that the two steps may be performed sequentially or simultaneously.

In the heat treatment section, one or more drying rolls (e.g. 30, 31, 32, 33) is heated to or slightly above the desired maximum internal web temperature. Pressure rolls 35, 36, 37, 38 are used to improve heat transfer between the drying rolls and the web, and preferably, these pressure rolls are also highly loaded to continue the high pressure densification step during heat treatment. The drying roll temperature necessary to achieve target web temperature is a function of several factors including web thickness, web moisture, web entering temperature, web speed, nip pressure, and roll diameter; its calculation is within the skill of the art. It is presently believed optimum in the case of linerboard from unbleached kraft pulp to achieve an internal web temperature of 550°F (289°C) and to maintain such temperature for two seconds or 465°F (240°C) in the case of paperboard from bleached kraft pulp or TMP board and to maintain this temperature for 60 seconds or 450°F (232°C) in the case of paperboard from SCMP and other sulfite pulp and to maintain this temperature for 5 seconds. In any event, the roll temperature must be at least 420°F (221°C) which is well in excess of the temperature of normal drying rolls. The heat treatment rollers are contained within an envelope 40, and air caps 41, 42, 43, 44 may be used to heat the web further as it passes over the rolls. An inert gas, steam or superheated steam atmosphere may be used for this purpose and to prevent oxidation or combustion at high temperatures.

Following heat treatment, the web may be passed over final rolls 50, 51 having air caps 60, 61 to condition the web, which is then calendered and reeled in a conventional manner.

The combined effect of high pressure densification and high temperature produce an unexpected combination of good wet strength and good folding endurance in the finished product.

The invention has been practiced as described in the following examples. The improvement in board quality will be apparent from an examination of the test results listed in the tables below.

EXAMPLE 1

Pine wood chips from the southeastern United States were cooked by the kraft process to an extent typical of pulp used in linerboard production. The cooked chips were converted to a pulp by passage through a disk refiner. The pulp was thoroughly washed with water to remove residual black liquor and was stored in the wet state at 38-42°F - (3°-6°C) in a refrigerator until sheets were prepared. The cooked, washed pulp had a kappa number of 98, indicating presence of 15% residual lignin and had a freeness of 720 ml by the Canadian Standard Freeness test, which values are typical of a pine linerboard pulp prior to beating.

A dispersion of the pulp in distilled water was converted to handsheets using a TAPPI sheet mold. The quantity of fiber in the dispersion was adjusted to give a TAPPI sheet weight of 3.6 g in the oven dried state, said weight being close to that of an air dried, 42 lb/1000 ft² (205 g/m²) commercial linerboard sheet. The sheets were wet pressed with blotters at 60 psi (415 kPa) prior to drying.

Three sets of sheets were prepared. Sheets from the first set were dried on TAPPI rings at room temperature according to TAPPI standard T205 om-81. This is a conventional (C) drying procedure. Sheets from the second set were also dried by the conventional procedure but this procedure was followed by a heat treatment (HT). The paper sheet was placed between two 150 mesh stainless steel screens, which assembly was placed in the platen press. Heat treatment was in accordance with the conditions found optimum for this invention, namely 2 seconds at 550°F (289°C) sheet internal temperature. To do this, single sheets were placed in a 550°F (289°C) Carver platen press for 4 seconds with 15 psi (105 KPa) as applied pressure. Previous experiments using a thermocouple buried in the sheet had shown that the sheet required 2 seconds to reach the target 550°F (289°C) temperature. Individual sheets from the third set were inserted in the wet state in a different platen press at 280°F (138°C). A pressure of 15 psi (105 KPa) was maintained for 5 seconds to dry surface fibers, after which the pressure was increased to 790 psi (5450 KPa) for 20 seconds. On completion of this press densification process - (PD) sheet moisture was about 10%. Each sheet was removed from the PD press and immediately placed in the other, HT press for 4 seconds at 550°F (289°C). All three sets of sheets were conditioned at 73°F (23°C) and 50% humidity for at least 24 hours before testing.

Fold, wet and conditioned tensile strength and conditioned compressive strength were the tests that were carried out. Wet tensile tests were carried

out immediately after excess water was blotted from test sheets which had been removed after 4 hours immersion in distilled water. Otherwise, this test was the same as the ASTM standard wet tensile test.

The results summarized in Table I show superior folding endurance and wet strength for the densified and heat treated sheets.

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TABLE I: COMPARISON OF PINE LINERBOARD HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES

Treatment	Density kg/m ³	Fold	Compressive Strength lb/in (KN/m)	Tensile Strength lb/in (KN/m)	Wet Tensile Strength lb/in(KN/m)
C	649	1714	31 (5.43)	73 (12.78)	2.7 (0.47)
C + HT	635	643	44 (7.71)	84 (14.71)	24.2 (4.24)
PD + HT	775	1115	44 (7.71)	94 (16.46)	26.0 (4.55)

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EXAMPLE 2

Hardwood chips from the southeastern United States were cooked by the kraft process to yield, after disk refining and washing, a 98 kappa pulp of

618 ml Canadian Standard Freeness. This pulp was mixed with the softwood of example 1 to give a mixture containing 60% softwood and 40% hardwood fiber. Sheets were prepared and tested following the procedure in Example 1. The superior fold and strength properties that were obtained are given in Table II.

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TABLE II: COMPARISON OF PINE/HARDWOOD LINERBOARD HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES

Treatment	Density kg/m ³	Fold	Compressive Strength lb/in (KN/m)	Tensile Strength lb/in (KN/m)	Wet Tensile Strength lb/in(KN/m)
C	546	831	25 (4.38)	57 (9.98)	2 (0.35)
C + HT	569	462	36 (6.30)	63 (11.03)	15 (2.63)
PD + HT	701	1032	39 (6.83)	73 (12.78)	17 (2.98)

EXAMPLE 2A

Pine wood chips were processed into a pulp as in Example 1, first paragraph. A dispersion of the pulp in distilled water was converted to handsheets using a Noble & Wood sheet mold. The quantity of fiber in the dispersion was adjusted to give a sheet weight of 7.9 g in the oven dried state. The sheets were wet pressed with blotters at 50 psi (346 kPa) prior to drying.

Three sets of sheets were prepared. Sheets from the first set were dried on a rotary drum dryer

in a conventional (C) manner. Sheets from the second set were heat treated (HT) as in Example 1, and sheets from the third set were densified and then heat treated (PD & HT) as in Example 1. One sample from each set was conditioned at 73°F, 50% relative humidity ("dry"); another sample was conditioned at 90°F, 90% relative humidity - ("moist"). Folding endurance, wet tensile strength and compressive strength tests were then carried out as in Example 1. The results, summarized below, show a marked improvement in both folding endurance and in tensile and compressive strength in high moisture conditions.

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TABLE IIA
COMPARISON OF PINE LINERBOARD HANDSHEETS
UNDER HIGH MOISTURE CONDITIONS

Treatment	Density kg/m ³	Fold	Compressive Strength		Wet Tensile Strength
			"Dry" lb/in KN/m	"Moist" lb/in KN/m	
C	412	441	17.7 (3.1)	10.2 (1.8)	1.8 (0.3)
C & HT	503	681	36.1 (6.3)	18.9 (3.3)	21.7 (3.8)
PD & HT	843	1878	37.6 (6.6)	24.0 (4.2)	27.1 (4.7)

EXAMPLE 3

The pine pulp used in Example 1 was subjected to three levels of beating by multiple passes through an Escher Wyss refiner to decrease the

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freeness of the pulp. Sheets were prepared and tested at each process level following the procedure in Example 1. The results in Table 3 again clearly demonstrate the lack of brittleness of the PD + HT sheets in comparison with sheets treated by the C + HT procedure.

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TABLE III: COMPARISON OF THE PINE LINERBOARD PULP
SHEET PROPERTIES AFTER 3 LEVELS OF BEATING

Canadian Standard Freeness (mls)	Treat- ment	Den- sity kg/m ³	Fold	Compressive Strength lb/in (KN/m)	Tensile Strength lb/in (KN/m)	Wet Tensile Strength lb/in (KN/m)
605	C	694	2037	38 (6.65)	80 (14.1)	3 (0.53)
605	C + HT	697	866	47 (8.23)	82 (14.36)	27 (4.73)
605	PD + HT	766	1315	48 (8.40)	85 (14.89)	30 (5.25)
505	C	753	2372	41 (7.18)	89 (15.58)	3 (0.53)
505	C + HT	737	625	50 (8.76)	88 (15.41)	31 (5.43)
505	PD + HT	770	1277	47 (8.23)	90 (15.76)	33 (5.78)
420	C	761	2536	40 (7.00)	89 (15.58)	3 (0.53)
420	C + HT	748	920	47 (8.23)	87 (15.23)	29 (5.08)
420	PD + HT	801	1117	50 (8.76)	94 (16.46)	38 (6.65)

These values may be compared to those shown in Table I, for unbeaten pulp (720 Canadian Standard Freeness).

EXAMPLE 4

On a conventional linerboard machine, three hard covered 12" diameter press nip rolls were located on drier cans #43, 45 and 47. Furnish of

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100% softwood kraft pulp was run on the machine and a 42 lb/1000 ft² (205g/m²) basis weight linerboard was obtained at a speed of 1550 ft/min. (473 m/min.). No nip pressure was applied to the nip rolls mentioned during the first stage of the trial and with conventional drying temperature, properties outlined below in Table IV were obtained. In the table, "MD" denotes testing along the machine length; "CD" denotes testing across the machine width.

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TABLE IV - CONVENTIONAL

Basis Weight	=	42 lb/1000 ft ² (205g/m ²)
Caliper	=	11.3 mils (.276mm)
Density	=	713 kg/m ³
Double Fold	MD =	2043
	CD =	1493
Compression Strength	MD =	39.1 lb/in (6.85 KN/m)
	CD =	21.9 lb/in (3.84 KN/m)
Dry Tensile	MD =	87.6 lb/in (15.3 KN/m)
	CD =	39.9 lb/in (6.99 KN/m)
Wet Tensile	MD =	10.1 lb/in (1.77 KN/m)
	CD =	4.8 lb/in (0.84 KN/m)

When this board was subject to high temperature treatment of 464°F for 30 seconds, properties shown in Table V were obtained. 15

TABLE V - HEAT TREATED

Basis weight	=	42 lb/1000 ft ² (205g/m ²)
Caliper	=	11.3 mil (.276 mm)
Density,	=	713 kg/m ³
Double Fold	MD =	15
	CD =	92
Compression Strength	MD =	48.0 lb/in (3.41 KN/m)
	CD =	19.6 lb/in (3.43 KN/m)
Dry Tensile	MD =	92.0 lb/in (16.11 KN/m)
	CD =	42.0 lb/in (7.36 KN/m)
Wet Tensile	MD =	36.0 lb/in (6.30 KN/m)
	CD =	17.1 lb/in (2.99 KN/m)

The increase in wet strength, coupled with the very great reduction in folding endurance, conform to prior art experience. To test the effect of densification, the press nip rolls were then activated. A

force of 230 pli (41 kg/cm) gave a nip pressure of 1225 psi (8445 KPa) and when three pressure nips were applied, the densified board gave test results as follows:

TABLE VI -DENSIFIED

Basis weight	=	42 lb/1000 ft ² (205g/m ²)
Caliper	=	10.5 mil (.266 mm)
Density	=	769
Double Fold	MD =	2025
	CD =	1244
STFI	MD =	42.3 lb/in (7.41 KN/m)
	CD =	23.6 lb/in (4.13 KN/m)
Dry Tensile	MD =	89.0 lb/in (15.59 KN/m)
	CD =	44.6 lb/in (7.81 KN/m)
Wet Tensile	MD =	18.2 lb/in (3.18 KN/m)
	CD =	10.7 lb/in (1.87 KN/m)

The densified board was then heat treated at 464°F for 20 seconds. The following results were obtained.

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TABLE VII - DENSIFIED AND HEAT TREATED

Basis weight	=	42	lb/1000 ft ² (205g/m ²)
Caliper	=	10.2	mil (.266 mm)
Density	=	789	
Double Fold	MD	=	1450
	CD	=	1142
STFI	MD	=	46.9 lb/in (8.21 KN/m)
	CD	=	26.1 lb/in (4.57 KN/m)
Dry Tensile	MD	=	92.0 lb/in (16.11 KN/m)
	CD	=	49.0 lb/in (8.58 KN/m)
Wet Tensile	MD	=	34.1 lb/in (5.47 KN/m)
	CD	=	17.7 lb/in (3.09 KN/m)

The unexpected lack of brittleness (as measured by the folding endurance test) of the densified and heat treated product (Table VII) when compared with the other high wet strength paperboard (Table V) can be identified as a direct result of the sequence of densification and high temperature treatment.

EXAMPLE 5

To illustrate the effect of densification prior to conventional or dynamic press drying, handsheets were prepared from a 60% softwood, 40% hardwood high yield pulp blend of the linerboard type. The sheets were divided into two main groups. The first group of sheets were wet pressed at an intensity level approximating that in a conventionally equipped production machine wet press (CWP). The second group were pressed at an intensity level approximating that of a modern production machine equipped with a shoe press (SP).

Each group of sheets was further subdivided into individual sheets which were retained for testing after drying on a steam-heated rotating drum,

or press drying by passage through the nip between a press roll and the rotating drum, or by static press drying between 150 mesh stainless steel screens at 465°F for 30 seconds with 15 psi pressure applied by means of a suitable press.

Heat treated control sheets which had been subjected to conventional wet pressing (CWP) and drying on the rotating drum had high caliper. Such thick sheets have minimal fiber-fiber contacting points. As adhesive forces develop at such points during drying, minimal contacting points result in poor folding endurance and wet tensile strength properties after heat treatment. Densification by use of the shoe press gave lower caliper and improved contact between fibers, and wet strength also increased. Dynamic press drying gave somewhat more efficient densification and provided a further improvement in wet tensile strength. The combination of shoe wet pressing and dynamic press drying provided further improvements after heat treatment. The final data in the table show what can be obtained by application of static press drying followed by heat treatment of sheets which had been subjected to the shoe pressing procedure.

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TABLE VIII

EFFECT OF DENSIFICATION ON FOLDING ENDURANCE AND WET
TENSILE STRENGTH OF 46 lb/ft² HEAT TREATED HANDSHEETS

<u>Process</u>	<u>Caliper (mils)</u>	<u>Density (kg/m³)</u>	<u>Double Fold</u>	<u>Wet Tensile Strength (lb/in)</u>
CWP - no HT drum dried w/o press	19.4	457	30	2.7
CWP - HT drum dried w/o press	19.19	445	198	11.3
SP - HT drum dried w/o press	13.3	665	543	12.3
CWP press dried, HT	11.1	797	631	12.5
SP press dried, HT	10.7	827	725	14.6
SP static press dried, HT	11.8	750	572	17.8

EXAMPLE 6

Pine wood chips from the southeastern United States were cooked by the kraft process to an extent typical of pulp used in linerboard production. The cooked chips were converted to a pulp by passage through a disk refiner. The pulp was bleached and washed with water to remove residual black liquor and was stored in the wet state at 38-42°F (3°-6°C) in a refrigerator until sheets were prepared. The cooked, bleached pulp contained substantially no lignin and had a freeness of 720 ml by the Canadian Standard Freeness test, which values are typical of a bleached pine pulp prior to beating.

A dispersion of the pulp in distilled water was converted to handsheets using a TAPPI sheet mold. The quantity of fiber in the dispersion was adjusted to give a TAPPI sheet weight of 3.6 g in the oven dried state, said weight being close to that of an air dried, 42 lb/1000 ft² (205 g/m²) commercial sheet. The sheets were wet pressed with blotters at 60 psi (415 kPa) prior to drying.

Three sets of sheets were prepared. Sheets from the first set were dried on TAPPI rings at room temperature according to TAPPI standard T205 om-81. This is a conventional (C) drying procedure. Sheets from the second set were also dried by the conventional procedure but this procedure was followed by a heat treatment (HT). The

paper sheet was placed between two 150 mesh stainless steel screens, which assembly was placed in the platen press. Heat treatment was in accordance with the conditions found optimum for this invention, namely 60 seconds at 465°F - (240°C) sheet internal temperature. To do this, single sheets were placed in a 465°F (240°C) Carver platen press for 60 seconds with 15 psi - (105 KPa) as applied pressure. Individual sheets from the third set were inserted in the wet state in a different platen press at 280°F (138°C). A pressure of 15 psi (105 KPa) was maintained for 5 seconds to dry surface fibers, after which the pressure was increased to 790 psi (5450 KPa) for 20 seconds. On completion of this press densification process (PD) sheet moisture was about 10%. Each sheet was removed from the PD press and immediately placed in the other, HT press for 4 seconds at 465°F (240°C). All three sets of sheets were conditioned at 73°F (23°C) and 50% humidity for at least 24 hours before testing.

Folding endurance and wet tensile strength were the tests that were carried out. Wet tensile tests were carried out immediately after excess water was blotted from test sheets which had been removed after 4 hours immersion in distilled water. Otherwise, this test was the same as the ASTM standard wet tensile test.

The results summarized in Table IX show superior folding endurance and wet strength for the density and heat treated sheets.

TABLE IX: COMPARISON OF BLEACHED PINE KRAFT PAPERBOARD HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES

<u>Treatment</u>	<u>Density kg/m³</u>	<u>Folding Endurance cycles</u>	<u>Wet Tensile Strength lb/in (KN/m)</u>
C	530	142	0.0 (0.00)
C + HT	523	62	3.7 (0.65)
PD + HT	766	391	5.5 (0.96)

EXAMPLE 7

A southern hardwood bleached kraft pulp in the never-dried state was processed in accordance with the procedure in Example 6. The test results illustrate the lack of wet pulp strength and the somewhat brittle nature of conventionally dried

hardwood pulp sheets. Heat treatment of the conventionally dried sheets produced rather mediocre wet strength accompanied by increased brittleness. However, sheets processed in accordance with this invention gave fold values improved by a factor of almost four, thereby demonstrating a pronounced lowering or brittleness in the sheets, which also had significantly improved wet strength.

COMPARISON OF BLEACHED HARDWOOD KRAFT PAPERBOARD HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES

<u>Treatment</u>	<u>Density kg/m³</u>	<u>Folding Endurance cycles</u>	<u>Wet Tensile Strength lb/in (KN/m)</u>
C	535	15	0.0 (0.00)
C + HT	530	5	3.4 (0.60)
PD + HT	652	57	6.1 (1.07)

Example 8 illustrates the process of this invention when applied to TMP board.

EXAMPLE 8

Pine wood chips from the southeastern United States were converted to a pulp by passage through a 250°F (121°C) disk refiner. The pulp was stored in the wet state at 38-42°F (3°-6°C) in a refrigerator until sheets were prepared.

A dispersion of the pulp in distilled water was converted to handsheets using a TAPPI sheet mold. The quantity of fiber in the dispersion was adjusted to give a TAPPI sheet weight of 3.6 g in the oven dried state, said weight being close to that of an air dried, 42 lb/1000 ft² (205 g/m²) commercial linerboard sheet. The sheets were wet pressed with blotters at 60 psi (415 kPa) prior to drying.

Three sets of sheets were prepared. Sheets from the first set were dried on TAPPI rings at room temperature according to TAPPI standard T205 om-81. This is a conventional (C) drying

procedure. Sheets from the second set were also dried by the conventional procedure but this procedure was followed by a heat treatment (HT). The paper sheet was placed in a 465°F (240°C) Carver platen press for 60 seconds with 15 psi (105 KPa) as applied pressure. Individual sheets from the third set were inserted in the wet state in a different platen press at 280°F (138°C). A pressure of 15 psi (105 KPa) was maintained for 5 seconds to dry surface fibers, after which the pressure was increased to 790 psi (5450 KPa) for 20 seconds. On completion of this press densification process (PD) sheet moisture was about 10%. Each sheet was removed from the PD press and immediately placed in the other, HT press for 60 seconds at 465°F (240°C). All three sets of sheets were conditioned at 73°F (23°C) and 50% humidity for at least 24 hours before testing.

Folding endurance and wet tensile strength were the tests that were carried out. Wet tensile tests were carried out immediately after excess water was blotted from test sheets which had been removed after 4 hours immersion in distilled water.

Otherwise, this test was the same as the ASTM standard wet tensile test.

The results summarized in Table X show superior folding endurance and wet strength for the densified and heat treated sheets.

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**TABLE XI. COMPARISON OF TMP PAPERBOARD
HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES**

<u>Treatment</u>	<u>Density kg/m³</u>	<u>Folding Endurance</u>	<u>Wet Tensile Strength lb/in(KN/m)</u>
C	377	0	0.00 (0.00)
C + HT	390	0	6.00 (1.05)
PD + HT	652	1	14.3 (2.50)

surface fibers, after which the pressure was increased to 790 psi (5450 KPa) for 20 seconds. On completion of this press densification process - (PD) sheet moisture was about 10%. Each sheet was removed from the PD press and immediately placed in the other, HT press for 60 seconds at 465°F (240°C). All three sets of sheets were conditioned at 73°F (23°C) and 50% humidity for at least 24 hours before testing.

Folding endurance and wet tensile strength were the tests that were carried out. Wet tensile tests were carried out immediately after excess water was blotted from test sheets which had been removed after 4 hours immersion in distilled water. Otherwise, this test was the same as the ASTM standard wet tensile test.

The results summarized in Table X show superior folding endurance and wet strength for the densified and heat treated sheets.

**TABLE X. COMPARISON OF TMP PAPERBOARD
HANDSHEETS AFTER THE C, THE C + HT AND PD + HT PROCEDURES**

<u>Treatment</u>	<u>Density kg/m³</u>	<u>Folding Endurance</u>	<u>Wet Tensile Strength lb/in(KN/m)</u>
C	377	0	0.00 (0.00)
C + HT	390	0	6.00 (1.05)
PD + HT	652	1	14.3 (2.50)

Example 9

A mixture of spruce and fir wood chips was cooked by the SCMP process to a yield of 92% by weight of dry chips. The cooked chips were converted to a pulp by passage through a disk refiner. The pulp was washed with water to remove residual cooking chemical and solubilized material. Latency removal was accomplished by stirring at 4% consistency for 20 minutes at 85-90°C. Pulp freeness was 705 ml by the Canadian Standard Freeness Test.

A dispersion of the pulp in distilled water was converted to handsheets using a TAPPI sheet mold. The quantity of fiber in the slurry fed to the

mold was adjusted to give a basis weight of 42 lb/1000 ft² (205 g/m²) in the oven dried state.

Two sets of sheets were prepared. Sheets from the first set were dried on TAPPI rings at room temperature after wet pressing. Wet pressing and drying were in accordance with the procedure in TAPPI T-205 om-81. Sheets from the second set were placed between two 150 mesh stainless steel screens and pressed in a platen press at 300 psi - (2067) kPa and 450°F (232°C) platen temperature for different times between 5 and 60 seconds. This drying procedure effectively combines the densification and heat treatment stages and is known as high temperature press drying (HTPD). All sheets were conditioned at 73°F (22.5°C) and 50% humidity for at least 48 hours before testing.

Folding endurance, wet tensile and conditioned tensile strengths were the tests that were carried out. Wet tensile tests were run immediately after excess water was blotted from test sheets which

had been removed after four hours immersion in distilled water. Otherwise, this test was the same as the ASTM standard tensile test for a conditioned sheet.

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**TABLE XI- TEST RESULTS COMPARING SCMP SHEETS
DRIED BY THE TAPPI PROCEDURE WITH HIGH TEMPERATURE
PRESS DRIED (HTPD) SHEETS**

Drying Procedure	Time in 232°C Press (secs)	Sheet Density (kg/M ³)	Conditioned Tensile	Wet Tensile	Double Fold
			Strength lb/in. (kN/m)	Strength lb/in. (kN/m)	
TAPPI	60	617	72.4(12.67)	8.95(1.565)	18
HTPD	20	645	72.6(12.70)	7.53(1.317)	14
HTPD	5	639	72.7(12.71)	6.50(1.137)	13
HTPD	--	389	40.1(7.02)	2.57(0.449)	9

The improved tensile properties, both wet and conditioned, and the lowered brittleness as illustrated by the increased number of double folds, are in accordance with the invention.

Example 10

A sample of a commercial low yield sulfite pulp in the never dried state, prepared from northern softwood chips, was obtained and converted to handsheets using a TAPPI mold. The quantity of fiber in the slurry fed to the mold was adjusted to give a basis weight of 42 lbs/1000ft² (205.5 kg/M²) in the oven dried state. Four sets of sheets were prepared and wet pressed as specified in accordance with the procedure in TAPPI T-205 om-81. Two of the four sets of sheet were dried on rings as required by the procedure. These sheets were considered to be dried by a conventional (C) method. One of the two sets of dry sheets was then subjected to heat treatment. For heat treatment, each sheet was placed between two 150 mesh stainless steel screens and inserted between the platens of a preheated platen press. Press temperatures of 392, 428, 454°F (200, 220 and 240°C) were studied. The platens were immediately closed and 15 psi (103.4 kPa) pressure was

25 applied for 5 seconds. Sheets were immediately removed from screens and allowed to cool after pressing.

Preliminary experiments using a thermocouple wire buried in the sheet showed the sheet internal temperature after 2 seconds is only 1-2°C lower than the platens temperature.

30 The third and fourth sets of sheets were placed between the 150 mesh screens and densified by a press densification (PD) procedure during the process of drying. To carry out the PD procedure, the wet sheets and the screens were placed between the platens of a second press and subjected to 15 psi (103.4 kPa) pressure at 138°C for 5 seconds to dry surface fibers, after which the pressure was increased to 790 psi (5443 kPa) for 20 seconds. On completion of this PD process, sheet moisture was about 10%. One set of sheets was retained for testing. Each individual sheet and screens from the second set were removed from the PD press and immediately placed in the other, HT press for 5 seconds. HT press temperatures of 200, 220 and 240°C were studied. HT pressure was 15 psi - (103.4 kPa). All sheets were conditioned at 22.5°C for at least 48 hours before testing.

50 Fold and wet tensile strengths were determined as specified in Example 8

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**TABLE XII EFFECT OF 5 SEC. 15 PSI (103.4 kPa)
HEAT TREATMENT AT THREE TEMPERATURES
ON SULFITE PULP HANDSHEET PROPERTIES**

Method of Drying	HT Temperature (°C)	Sheet Density (kg/M ³)	Double Fold	Wet Tensile Strength lb/in. kN/m
C	---	702	979	2.86 0.500
PD	---	847	1467	5.09 0.891
C + HT	200	701	521	5.75 1.006
PD + HT	200	856	1337	7.56 1.323
C + HT	220	699	258	9.39 1.643
PD + HT	220	833	1038	13.13 2.300
C + HT	240	696	117	12.33 2.158
PD + HT	240	834	457	17.39 3.042

The results show that wet strength improves as heat treatment temperature is increased. Fold decreases as heat treatment temperature is increased, but the decrease is much less pronounced for the densified, heat treated sheets. This shows that the densified sheets are much less brittle than the conventional sheets, even after heat treatment to yield enhancement of wet strength.

Claims

1. A method of producing paper products with improved wet strength from bleached or unbleached kraft pulp, thermomechanical pulp, SCMP or sulfite pulp, while preserving its folding endurance, comprising steps of

subjecting paper to high pressure densification during its production, and

heating the paper so as to raise its internal temperature to at least 420°F (216°C) for a period of time sufficient to increase the wet strength thereof.

2. The method of claim 1, wherein said densification and heating steps are carried out simultaneously.

3. The method of claim 1, wherein said densification step precedes said heating step.

4. The method of claims 1 to 3, wherein said internal temperature is in the range of 420°F (216°C) to 572°F (300°C).

5. The method of claims 1 to 4, wherein the kraft pulp is unbleached and the internal temperature is about 550°F (289°C), and when the pulp is bleached, or when the pulp is thermomechanical pulp, the internal temperature is about 485°F (240°C) and when the pulp is SCMP or sulfite pulp, the internal temperature is about 450°F (232°C).

6. The method of claims 1 to 5, wherein in said densification step the paperboard is compressed to a density of 600 to 1200 kg/m³.

7. The method of claims 1 to 6, wherein said densification includes applying sufficient pressure to the paper to produce density in range of 700-900 kg/m³ prior to said heating step.

8. The method of claims 1 to 7, wherein said paper prior to said densification step, has a moisture content in the range of 10% to 70% by weight.

9. The method of claims 1 to 8, wherein said paper product is linerboard or paperboard.

10. The method of claim 9, wherein said paper product is linerboard and has a basis weight in the range of 125 to 464 g/m².

11. The method of claims 1 to 9, wherein said paper product is paperboard and has a basis weight in the range of 30 to 464 g/m² and when the paperboard is produced from thermomechanical pulp, the basis weight is 60-464 g/m².

12. A linerboard of high wet strength and high folding endurance, producible with the method according to any of claims 1 to 11.

13. A linerboard as in claim 12, having a wet strength of at least 15 lb/in, and satisfying a folding endurance test of at least 300 cycles, preferably at least 1000 cycles.

14. A linerboard as in claim 12, from SCMP or sulfite pulp, having a wet strength of at least 6 lb/in, and satisfying a folding endurance test of at least 10 cycles.

15. A bleached kraft paperboard of high wet strength and high folding endurance, producible with the method according to any of claims 1 to 9 and 11.

16. A bleached kraft paperboard as in claim 15, having a wet strength of at least 5 lb/in, and satisfying a folding endurance test of at least 50 cycles.

17. A bleached kraft paperboard as in claim 15, having a wet strength of at least 15 lb/in, and satisfying a folding endurance test of at least 300 cycles.

18. A paperboard of high wet strength and high folding endurance, producible from thermomechanical pulp with the method according to any of claims 1 -9 and 11.

19. A paperboard as in claim 18, having a wet strength of at least 10 lb/in, and satisfying a folding endurance test of at least one cycle.

20. An apparatus for carrying out the method according to any of claims 1 to 11.

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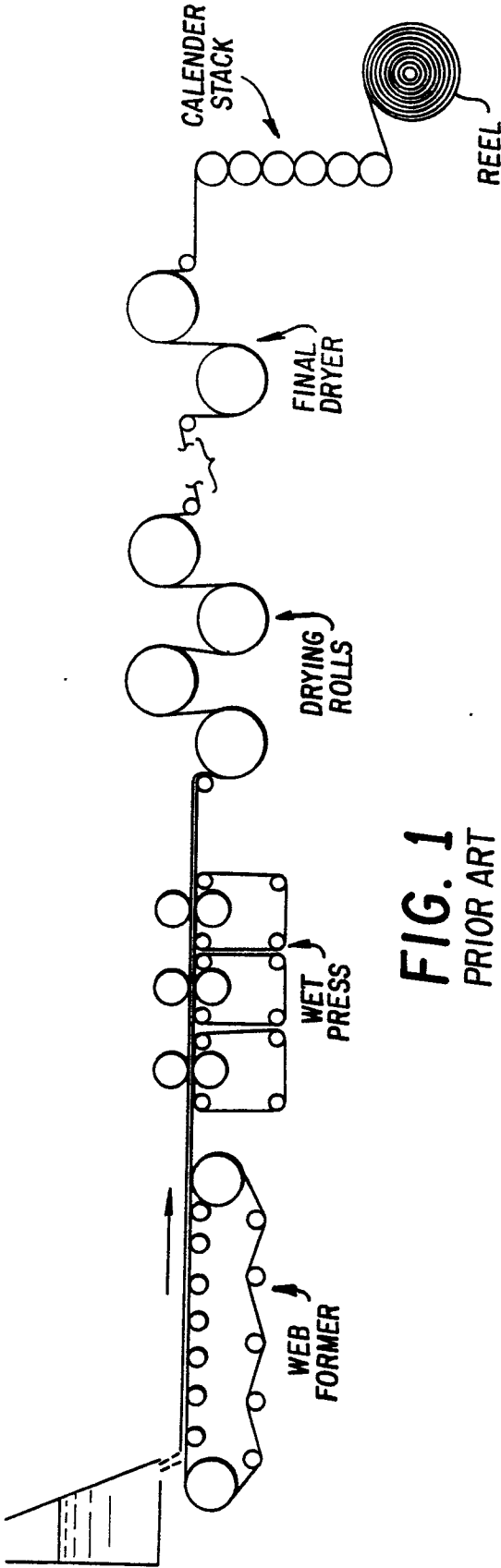


FIG. 1
PRIOR ART

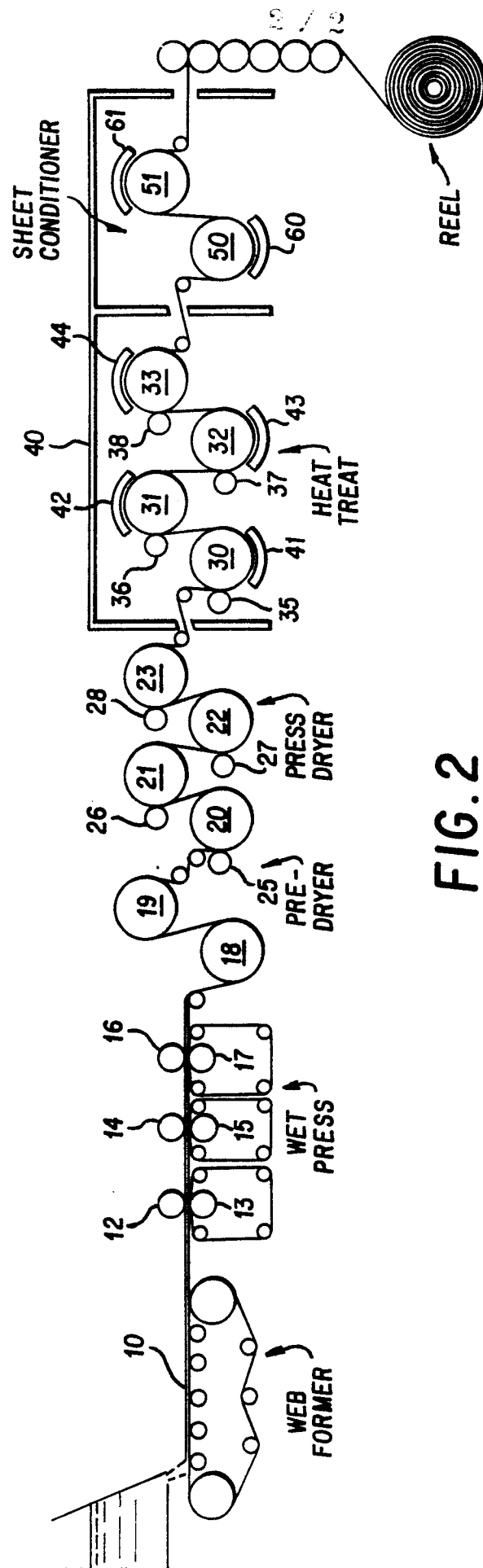


FIG. 2



DOCUMENTS CONSIDERED TO BE RELEVANT			EP 86111513.7
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	AT - B - 221 923 (LIPPKE) * Totality * -----		D 21 F 11/02
			TECHNICAL FIELDS SEARCHED (Int. Cl.4)
			D 21 F
The present search report has been drawn up for all claims			
Place of search VIENNA		Date of completion of the search 19-11-1986	Examiner HOFMANN
CATEGORY OF CITED DOCUMENTS			
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	