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# (54) ABSORBENT PAPER SHEET MARKED WITH PATTERN SIMULATING WATERMARK AND PROCESS FOR THE MARKING THEREOF

MIT EINEM WASSERZEICHENÄHNLICHEN MUSTER VERSEHENES SAUGFÄHIGES PAPIER UND VERFAHREN ZUM VERSEHEN DES PAPIERS MIT MUSTERN

TISSU-PAPIER MARQUE D'UNE EMPREINTE SIMULANT UN FILIGRANE ET PROCEDE DE FABRICATION ASSOCIE

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• •	Inventors: LEFEBVRE DU GROSRIEZ, Carol F-68230 Turckheim (FR)		WO-A-94/06623 US-A- 3 486 970 US-A- 4 847 118	GB-A- 2 056 881 US-A- 4 166 758 US-A- 4 945 932

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#### Description

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**[0001]** The present invention relates to a sheet of absorbent paper made of tissue paper, comprising one or more plies, each having a basis weight of approximately 12 to approximately 70 g/m<sup>2</sup>, and a part of the surface of which is marked. It also relates to a marking process in order to obtain this sheet.

**[0002]** The invention finds application in sanitary and domestic papers, such as toilet paper, papers for wiping, such as kitchen paper towel, disposable handkerchiefs, facial tissues or else products of the table range, such as disposable napkins and tablecloths. The invention allows finishing the tissue paper products.

[0003] The term « marking », as it is used in the following description, consists in compressing certain parts of one side of the sheet, thus forming compacted areas and significantly reducing the thickness of the sheet in these areas without forming a corresponding relief on the opposite side.

**[0004]** In order to create a pattern on a sheet of absorbent paper made of tissue paper, of the type comprising several plies, and to bond the latter together, it is common to use the embossing technique which makes it possible to create, on part of the surface of each of the plies forming the sheet, reliefs with corresponding depressions on the opposite

- <sup>15</sup> side of the plies. As a result, the sheet which has been embossed has increased in thickness. In order to bond the plies together, an adhesive is often used. A pre-embossed first ply receives, via a sizing device, an adhesive on the end of the protuberances produced during the embossing operation. This ply is then brought into contact with another ply, also pre-embossed, in order for the two plies to be adhesively bonded together, connecting them at the contact points or surfaces generated by the embossing. The use of an adhesive makes the process particularly tricky and, in
- 20 fact, it is necessary to take care that the phenomenon of filling does not occur in the conversion installation. Moreover, the adhesive stiffens the sheet in the bonding areas. The fact of embossing and of creating reliefs increases the roughness of paper surface. Conventional embossing also adversely affects the mechanical properties of strength and elongation and increases the thickness of the structure.
- [0005] Finally, the embossing technique limits the choice of patterns which can be applied on the sheet of absorbent paper. The reason for this is that only patterns which are rounded and devoid of sharp angles may be used to engrave the embossing surfaces so as in particular to avoid too great a loss of mechanical strength.
  [0006] There is another way of barding the plice without using adhesing by emboding the plice between a first strength.

**[0006]** There is another way of bonding the plies without using adhesive, by embossing the plies between a first engraved roll and a paper receiving roll or a steel receiving roll having female patterns or impressions of the patterns engraved on the first roll. This technique is applied, for example, in order to bond the edges of folded handkerchieves and of table napkins.

**[0007]** The publication of international patent application WO-A-95/27429 discloses a cellulose cloth for hygiene comprising at least two layers of tissue with an embossed pattern consisting of a plurality of individual spot-shaped impressions which deform and mutually transversely connect the layers of tissue. The impressions are formed by embossed spots which originate from the two outer layers of tissue and are curved concavely inwardly in each case

- <sup>35</sup> and which are mutually transversely aligned and in whose head-to-head base regions the tissue layers are connected to one another by the embossing action. The impressions are applied on both sides. The embossing process requires an appropriately precise arrangement and control of the rigid embossing rollers in such a way that the embossed mounds with which the external surface of the embossing rollers are provided, are arranged exactly head-to-head in each case but do not touch one another. This process might require a high level of precision and control.
- <sup>40</sup> **[0008]** Marking, simply by deforming under stress, without using adhesive, also exists and is termed « press marking ». This technique consists in deforming, under a stress and when cold, a sheet of absorbent paper between an engraved roll made of steel and a smooth receiving roll also made of steel. The marking is performed in the form of bands by means of « roller-wheels » which form very small compressed surfaces in the contact areas during the deformation. However, a drawback is observed with respect to the pattern geometry, which is imperfect. This drawback
- <sup>45</sup> results from the localization of the pattern, limited to a band parallel to the edge of the product, combined with difficulty in guiding the sheet during press marking. In addition, this technique enables only the edges of a sheet to be bonded together and not the surface of the sheet over its entire width.

**[0009]** The choice of patterns is limited to the use of very small surfaces in the form of discs or diamonds, akin to points. A major drawback encountered in this type of marking is the lack of uniformity in the marking itself. The pattern is not permanent and is irregular especially because of the wear of the roller wheels, in particular in the case of the

<sup>50</sup> is not permanent and is irregular, especially because of the wear of the roller-wheels, in particular in the case of the press marking of toilet paper. In addition, it is not easy to see the pattern by holding it up to the light, unlike the water-marked designs which are mentioned in the following description.

[0010] Endeavours have been made to develop marking processes enabling the shape of the patterns to be varied freely. In particular FR-A-2 672 843 describes a process which consists in simultaneously marking patterns of any shape, in particular patterns having sharp angles, and adhesively bonding the plies. However, this process, even by reducing the amounts of adhesive applied, does not dispense with using it altogether. Moreover, the pressure exerted between the two engraved and undeformable steel rolls is relatively low and does not run the risk of tearing the sheet. The two rolls must be separated from each other by a distance barely less than the thickness of the two plies. There

is also a limitation in the patterns as regard to their height and to the spacing of the protuberances on the roll, in order to enable correct application of the adhesive to the plies in the region of the protuberance without applying adhesive to those parts of the plies which lie between the protuberances. This process therefore results in certain constraints.

- [0011] Watermarks have been used to mark papers, generally in fields quite far removed from those of sanitary and domestic papers. A watermark is the impression of a design in the thickness or body of the paper, which is produced intentionnaly in the paper, and is visible when viewed against a contrasting background. Watermarking is a very ancient technique and has served since the XIIth and XIIIth centuries to mark sheets, to trace their movement in the market place and to vouch for their provenance. Currently, watermarks are more conventionally used for papers of low grammage and thickness. They are encountered on paper of the uncoated type, for example letter paper. It is usually applied
- <sup>10</sup> on thin papers, such as cigarette paper having a basis weight of the order of 10 to 23 g/m<sup>2</sup> and which partly contains textile fibres (hemp and flax fibres), « bible » papers having a basis weight of approximately 25 to 36 g/m<sup>2</sup> and on special papers such as bank notes or filter paper. Those papers are different from tissue paper and are illustrated by prior patent literature.
- [0012] The watermark is termed a « wet » or « true » watermark, when it is produced during the manufacture of the paper in the wet phase of the process, either by means of an impression in the form of reliefs or in the form of depressions in the wire, for example, of the wire mould or of the cylinder mould machine, or by means of an impression in the form of reliefs or depressions at the surface of an open-ended cylinder rotating in contact with the wet pulp on the forming wire of a Fourdrinier former. This technique is used for the manufacture of bank notes. The design is then obtained by localized displacement of the fibers.
- 20 **[0013]** US-A-5,328,565, without mentioning watermarks, describes an absorbent paper made of tissue paper comprising a single ply and including surfaces with a non-embossed pattern. This pattern produces regions in the paper having different opacity, height and creping frequency (the number of times a peak appears on the surface for a given distance). The various visually discernible regions forming a particular pattern have different basis weights.
- [0014] The pattern is formed directly by the wire during the drying by means of the blowing of air through the sheet in the wet state. After drying, a pattern is certainly formed on the sheet but it does not stand out in contrast with the other areas since the areas with patterns are not actually and clearly visible when viewed against a contrasting background, as conventionally the watermarked patterns are, and these areas do not have a smooth and glossy appearance. [0015] The watermark is called a rubber mark (one still speaks of press marking) when the impression is produced on the paper during its manufacture on the machine by passing the wet web through a roller-whell press.
- <sup>30</sup> **[0016]** An imitation or simulated watermark is an impression similar in appearance to a true watermark, produced in the finished paper by mechanical means (for example the impression produced on cigarette paper). In this case, the fibres are crushed and compacted. Some patents describe this kind of watermark for papers different from the absorbent tissue paper.

**[0017]** US-A-4,945,932 reveals the impression of an imitation watermark on a cigarette paper by the means of a filigree calender.

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**[0018]** Another example is given by US-A-4,166,758 disclosing a matted transparent paper for impression which is obtained by moistening a paper and subjecting it to a calendering treatment with the use of a heated embossing metal roll having a finely engraved surface with sharp reliefs.

[0019] The simulated watermark may also be produced by removing material or else by applying certain products, such as resin-based substances. Many techniques for producing a simulated watermark on small quantities of finished paper have been developed for replacing the wet watermarking which requires much heavier and more expensive installations.

**[0020]** EP-A-0,195,887 describes a process for producing a watermarking resembling a wet watermark, which means that the paper documents obtained in this way cannot be falsified. This process consists in passing a sheet of paper

- <sup>45</sup> between two rolls rotating in opposite directions, one of the rolls including a pattern and being made of steel. The pattern thus marked is visible when the sheet is held up to the light or by difference in reflection of light on the paper. [0021] CA-A-1,116,851 relates to the surface treatment of a sheet of paper in order to create a pattern by removal of material. The process here consists in passing the sheet of paper between a roll which includes marking patterns and an abrasive roll or else a roll provided with cutters, the two rolls rotating at different speeds. The abrasive material
- <sup>50</sup> or the cutters remove from the sheet of paper a constant thickness of approximately 1/3 to 2/3 of the total thickness of the sheet. Thus, a simulated watermark is obtained which has a light transmission which is different from that of those parts of the sheet which have not undergone removal of material. Papers which have undergone this treatment are also unfalsifiable.
- [0022] The simulated watermark may also be produced by impregnating a resin-based composition into a sheet of paper, which makes the sheet translucent in the impregnation areas. This technique and compositions which can be used are widely described in the patent literature, for example GB-A-1,489,084 and US-A-3,985,927, No. 4,824,486 and No. 4,919,044.
  - [0023] Another example of simulated watermark is described in EP-A-0,203,499. This entails a process which con-

sists in preparing a sheet of paper containing a suitable amount of a heat-sensitive material, a polymer of the polyethylene type, and in applying heat to part of the surface of the sheet so as to make that part of the surface of the sheet translucent.

- **[0024]** The subject of International Application WO-A-94/07694 is a device for making a simulated watermark on paper (letterhead paper, certified documents, etc.) by chemical means. The simulated watermark is made and the paper is printed preferably in one and the same step. More precisely, this device comprises the combination of coating rolls applying the chemical composition serving for making the simulated watermark with a roll provided with a matrix and an impression cylinder.
- [0025] For another, different, type of paper, such as filter paper which contains cellulosic fibres and synthetics fibres, it is known to create a repeating pattern such as a semi-perforation at regular intervals. The process coonsists in applying a series of fluid jets and then in heating in order to set the synthetic fibres. EP-A-0,446,138 relates more precisely to a process for manufacturing filter bags which consists, after producing, using a known technique, a nonwoven paper composed of a layer based on synthetic fibres and of a layer of cellulosic fibres which are superimposed, in subjecting the non-woven paper to a calendering operation between a support structure, a smooth roll or a roll having an elastic pliant external surface, and a heated roll having reliefs
- <sup>15</sup> an elastic pliant external surface, and a heated roll having reliefs. [0026] In a similar application teabags made of paper (in this case a paper consisting of long fibres or else of non-woven material), the subjects of International Patent Application WO-A-94/06623 is a process for producing a pattern which is in the form of substantially transparent areas. The prior art in this field also uses the technique of creating perforations using jets. The novel process according to this international application consists in passing a sheet of
- 20 paper through a heated calender while applying a liquid, a synthetic material such as polypropylene, which allows the enhancement of the embossing effect and limits the loss of strength in the marked areas.
  [0027] The present invention relates to the application of a pattern or impression resembling a watermarked pattern, to a sheet of absorbent paper made of tissue paper, when that sheet has already been manufactured and dried; which process has hitherto never been employed. The watermark applied to the sheet of absorbent paper according to the
- 25 invention belongs to the category of imitation or simulated watermarks produced on finished paper using mechanical means; and the term « watermark » used hereafter in the following description of the invention has the above-mentioned meaning.

**[0028]** The object of the invention is to provide a sheet of absorbent paper, made of tissue paper, which mitigates all the drawbacks mentioned above for sheets of paper of this same kind which have been marked or embossed by processes in the prior art.

**[0029]** A further object of the invention is to provide a sheet of absorbent paper which includes an aesthetic pattern without modifying the thickness of the sheet by removal of material; or without modifying its mechanical and physical properties; and in particular without adversely affecting the softness properties of the sheet. The invention allows an infinitude of designs to be chosen for the patterns. It provides a matt/gloss contrast effect on the sheet : the marked

<sup>35</sup> parts are smooth and glossy and the non-marked parts remain matt and « fluffy ». The contrast effect can be correlated with the reflection of light. The invention also provides a translucent effect ; the marked parts being visible when viewed against a contrasting background.

**[0030]** In the case of a sheet comprising at least two plies, the object of the invention is also to connect the two plies together easily.

40 [0031] Another object of the invention is to provide a process for marking a sheet of absorbent paper made of tissue paper, based on short papermarking fibres, which has neither the problems of the conventional marking processes nor those of the known processes, mentioned above, for watermarking other types of paper; the watermarking process according to the invention does not use, for example synthetic components. The process according to the invention makes it possible to mark the sheet over its entire width unlike the known processes of press marking and of embossing

of edges without adhesive bonding by means of steel/steel or steel/paper rolls.
 [0032] The subject of the invention as described in claim 1 is a sheet of absorbent paper made of tissue paper comprising one or more plies, each having a basis weight of approximately 12 to approximately 70 g/m<sup>2</sup> and part of the surface of which is marked.

**[0033]** According to an essential characteristic of the invention, that part of the said surface which is marked is distinguished by an impressed pattern simulating a watermark,

- said impression being applied to one side of the sheet, the opposite side corresponding to said impression being plane,
- the marked surface being smooth, glossy and translucent, and

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 the marked surface comprising at least one marked unit region wherein the ratio of the thickness to the area of this marked unit region is less than 0.027 mm/mm<sup>2</sup>.

[0034] According to an advantageous characteristic of the invention, with regard to the light transmission through

the sheet, the difference between the average grey level of the marked surface and the average grey level of the unmarked surface is at least 20 by image analysis.

**[0035]** According to an other advantageous characteristic, the sheet comprises at least two plies joined together at the impression resembling the watermarked pattern.

- <sup>5</sup> **[0036]** The subject of the invention as described in claim 6 is also a process for marking with an impressed pattern resembling a watermark, a surface part of an absorbent tissue paper sheet comprising one or more plies each having a basis weight of approximately 12 to approximately 70 g/m<sup>2</sup>, the marked surface part comprising at least one marked unit region wherein the ratio of the thickness of said marked unit region to the area of said marked unit region is less than 0.027 mm/mm<sup>2</sup>, said process consisting in impressing one side of said sheet without forming a corresponding
- relief on the other opposite side, by deforming the said sheet under stress, by at least one pass between a hard engraved roll and a receiving roll having a surface hardness of more than Shore-D 40, the specific pressure exerted on the marked surface being at least 14,7 MPa (150 kg/cm<sup>2</sup>) and the linear marking speed being at least 50 m/min. [0037] According to an advantageous characteristic of the invention, the hardness of the receiving roll is at least Shore-D 80 and preferably greater than Shore-D 90.
- <sup>15</sup> **[0038]** According to an advantageous characteristic of the invention, the said engraved roll, preferably made of steel, is heated to a temperature ranging from approximately 50°C to approximately 100°C and preferably from approximately 75°C to approximately 85°C.

**[0039]** According to another characteristic of the invention, the process comprises a preliminary step of treating the sheet before deformation, consisting in bringing the surface of the sheet into contact with small amounts of water in the form of liquid or steam.

**[0040]** Other characteristics and advantages of the invention will appear more clearly on reading the following description with reference to the drawings in which :

- Figure 1 represents the surface of a sheet comprising simulated watermarks according to the invention;
- Figure 2 represents a cross-section of the sheet shown in Figure 1 along the line II-II;

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- Figures 3A, 3B and 3C represent marked and embossed patterns of the art and impressed patterns according to the invention, respectively;
- Figure 4 is a schematic diagram of an embodiment of the process according to the invention;
- Figure 5 represents a device which is used according to one embodiment of the process of the invention;
- Figure 6 represents a device which is used according to another embodiment of the process of the invention ; and
- Figure 7 represents a device which is used according to another embodiment of the process of the invention.

**[0041]** According to the invention, a part of the surface of the sheet is marked by an impressed pattern simulating a watermark.

- <sup>35</sup> [0042] The impression is applied to one side or the two sides of the sheet. The opposite side corresponding to the applied impression is plane. The impression does not form a relief on the corresponding opposite side of the sheet.
   [0043] If an impression is applied to the two sides of the sheet, no impression is applied to the opposite side corresponding to the side on which an impression is applied.
- [0044] Referring to Figure 1, a sheet 1 of absorbent paper made of tissue paper includes decorative patterns 2 and 3. These patterns produced in the sheet are simulated watermarks. They are marked in the thickness of the sheet and the lines 5 and 5', or the marking points defining them, are smooth, glossy and translucent to that the patterns are clearly visible when the sheet is viewed against a contrasting background. These lines 5 and 5' form an integral part of the decorative pattern. The papermarking fibres are crushed in the region of the marked surfaces. The pattern is also visible by the impression of relief which it gives the parts of the sheet surrounding the pattern, because of the
- 45 crushing. The sheet is also given a matt/gloss effect. The surface parts marked are smooth and glossy and the unmarked remaining parts keep the matt and more fluffy appearance of a conventional non-converted sheet of tissue paper. The marked surface is smooth due to the creped tissue paper sheet elevations which have been reduced or even removed by marking. This smooth and glossy aspect advantageously brings out the design of the pattern, enhancing the aesthetic appearance of the sheet. Any design, whether of simple or complex shape, may be envisaged,
- <sup>50</sup> such as flowers, for example a lotus flower, or calligraphed words or combinations of lines, strokes and curves defining a pattern. This pattern may occur just once, or more usually, it is repeated.
  [0045] The sheet may comprise one or more plies. Each ply has a basis weight lying within the range of approximately 12 to approximately 70 g/m<sup>2</sup>. The basis weight preferably lies within the range of approximately 12 to approximately 50 g/m<sup>2</sup>.
- <sup>55</sup> **[0046]** The density of the pattern, or alternatively the ratio of the marked surface area to the total surface of the sheet, is generally less than 30 per cent.

**[0047]** Figure 2 shows a cross-section of the sheet 1 comprising two plies 6 and 7. The thickness of the sheet is reduced at the place where the marked surface corresponding to the line 5' of the pattern of figure 1 is.

**[0048]** The marked surface area comprises at least one marked unit region fullfilling the following requirement. The ratio of the thickness of the marked unit region to the area of the marked unit region is less than 0.027. In other words, the marked surface of the sheet comprises at least one closed impression pattern having a reduced thickness and a minimal surface area. This minimal surface area corresponds to a marked unit region.

<sup>5</sup> **[0049]** According to figure 3, the patterns 3A of the art limited to a band of diamonds or dashes, parallel to the edge of the sheet, are obtained by « press marking » of a cold and dry sheet of tissue paper. The patterns  $3B_1$  and  $3B_2$  of the art illustrate embossed flowers for toilet tissue. The patterns  $3C_1$  and  $3C_2$  represent a tulip and a stylized flower, respectively, and illustrate a simulated watermark pattern according to the invention.

**[0050]** Samples of tissue paper sheet have been made with the corresponding patterns 3A, 3B, and 3C according to the marking or embossing process corresponding thereof.

**[0051]** All the sample sheets of the art comprise two plies. The sample sheets of the invention comprise one, two or three plies.

**[0052]** Patterns 3B and 3C have been splitted up into unit patterns (petals, stems, and leaves) to measure their corresponding unit marked (or embossed) surface areas and adding them to obtain the total pattern marked surface area.

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**[0053]** The following table illustrates the thickness and surface area of an impressed pattern (3C) according to the invention compared with conventional marked and embossed patterns (3A and 3B).

Patterns	3A	3B	3C
Thickness of unmarked surface (mm)	0.33	0.22 - 0.28	0.16 - 0.23
Thickness of marked surface (mm)	0.04 - 0.05	0.055 - 0.11	0.027 - 0.055
Thickness reduction	85 - 88 %	50 - 80 %	65 - 88 %
Pattern marked area (mm <sup>2</sup> )		66 - 70	59 - 79
Average unit marked surface (mm <sup>2</sup> )	1 - 1.15	10 - 16.5	7 - 16
Marked surface / total surface	< 5 %	> 5 %	< 5 %
R =	35 x 10 <sup>-3</sup> - 50 x 10 <sup>-3</sup>	33 x 10 <sup>-4</sup> - 110 x 10 <sup>-4</sup>	17 x 10 <sup>-4</sup> - 79 x 10 <sup>-4</sup>

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**[0054]** In view of the above table, the thickness reduction is similar between the embossed patterns (3B) and the simulated watermark patterns (3C). However, the average unit marked surface area of the « press marking » samples 3A is clearly smaller than the average unit marked surface area of the simulated watermark patterns (3C).

- <sup>35</sup> **[0055]** According to the invention, the simulated watermark pattern distinguishes over the « press marking » pattern by comprising at least one marked unit surface having a ratio of the thickness thereof to the area thereof of less than 0.027 mm/mm<sup>2</sup>. In other words, the simulated watermark pattern can comprise marked unit surfaces with a ratio less than 0.027 mm/mm<sup>2</sup> and other marked unit surfaces with a ratio greater than 0.027 mm/mm<sup>2</sup> such as small patterns like diamonds, circles, dashes, etc.
- <sup>40</sup> **[0056]** The simulated watermark patterns have marked unit surfaces with a ratio which is close to the embossed pattern one, but the obtained sheet is fundamentally different since the embossing technique creates, on part of the surface of each of the plies forming the sheet, reliefs with corresponding depressions on the opposite sides of the plies whereas the simulated watermark pattern corresponds to an impression applied to one side of the sheet, the opposite side of the sheet being plane without corresponding depressions.
- <sup>45</sup> **[0057]** The advantage of the simulated watermark pattern is to provide a decorative pattern similar to an embossed pattern with regard to the design, the size thereof and so on, with in addition the simulated watermarked effect.

**[0058]** A further advantage of the simulated watermark pattern is an unlimited choice of decorative patterns without technical restraints, compared with the known « press-marked » or embossed patterns.

**[0059]** The marked surface of the sheet is visible when viewed against a contrasting background. The marked surface is translucent.

- <sup>50</sup> **[0060]** Paper tissue sheets according to the invention, comprising impressed patterns simulating a watermark, have been analyzed with regard to light transmission. The method consists of providing a lighting through a sheet sample by the means of optical fibers and a light diffuser, measuring the grey level of an image by the means of a camera 3 CCD and of a QUANTIMET 600 S LEICA as an image analyzer apparatus.
- [0061] The following lighting parameters : luminosity, contrast, etc... are adjusted.
- **[0062]** To have an image, a surface is delimited. The image is divided into pixels which correspond to the image units. Each pixel of that surface has a grey level. The average value of all the pixels of the image gives the average grey level of a surface.

[0063] According to the grey scale, black has a grey level of zero and white has a grey level of 256.

**[0064]** The average grey level of a marked surface is measured by delimiting a small closed marked surface area. The average grey level of an unmarked surface is measured by delimiting a corresponding unmarked surface area closed to the selected marked one. And the difference between the two average grey levels is made.

[0065] This method is reproducible.

#### Controls:

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[0066] First tissue paper sheet samples (I) have patterns resulting from the wire during the drying of the sheet in the wet state. The papermarking fibers are probably displaced and the pattern regions have different opacity. The sheet samples (I) comprise two plies and have a total basis weight of approximately 40 g/m<sup>2</sup>.

**[0067]** Second tissue paper sheet samples (II) have embossed pattern corresponding to the pattern  $3B_2$  of figure 3. The sheet samples (II) also comprise two plies and have a total basis weight of approximately  $32 \text{ g/m}^2$ .

[0068] Third tissue paper sheet samples (III) have an embossed pattern corresponding to the stylized flower pattern  $3C_2$  of figure 3. These samples comprise three plies and have a total basis weight of approximately 52 g/m<sup>2</sup>.

#### Examples according to the invention

#### Example 1

**[0069]** A tissue paper sheet according to the Figure 1 of the invention, is made. This tissue paper sheet comprises two plies and has a basis weight of approximately 30 g/m<sup>2</sup>. Samples corresponding to the calligraphic LOTUS pattern

### <sup>25</sup> Example 2

**[0070]** A tissue paper sheet according to the invention with the stylized flower pattern of Figure  $3C_2$  is made. This tissue paper comprise two plies and has a basis weight of approximately 30 g/m<sup>2</sup>.

(1a), the lotus flower pattern (1b) and the stroke pattern (1c), have been analysed by image analysis, respectively.

#### 30 Example 3

**[0071]** A tissue paper sheet with the same pattern as the one of example 1 is made except that it comprises three plies and has a basis weight of approximately  $52 \text{ g/m}^2$ . The samples corresponding to the calligraphic LOTUS pattern, the lotus flower pattern and the stroke pattern are numbered 3a, 3b and 3c, respectively.

#### Example 4

**[0072]** A tissue paper sheet with the same pattern as the one of example 2 is made except that is comprises three plies and has a basis weight of approximately  $52 \text{ g/m}^2$ .

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Example 5

**[0073]** A tissue paper sheet according to invention with a tulip pattern of figure  $3C_1$  is made. It comprises three plies and has a basis weight of approximately 52 g/m<sup>2</sup>.

<sup>45</sup> **[0074]** The average grey level is determined for each marked and unmarked surface of every control and example sample.

**[0075]** The differences between the two grey levels of each control (I, II or III) and each example are listed in the following table.

5	n	1
v	v	

CONTROLS	I	II	Ш
Average grey level difference	10	5	0

Examples	Ex 1a	Ex 1b	Ex 1c	Ex 2
Average grey level difference	61	54	67	20

Examples	Control III	Ex 3a	Ex 3b	Ex 3c	4	5
Average grey level difference	0	72	52	83	30	41

**[0076]** In view of the above table, same pattern obtained by embossing (control III) and by simulating watermark (example 4) does give none or an average grey level difference, respectively. The light cannot go through the control III with an embossed pattern.

**[0077]** The embossed tissue paper does not allow the light to make the pattern stand out as a simulated watermark does.

**[0078]** A low average grey level difference is observed for control I the pattern of which resulting from a wet end papermaking process.

**[0079]** Higher average grey level differences are obtained with simulated watermarks : examples 1a, 1b, 1c, 2, 3a, 3b, 3c, 4 and 5.

**[0080]** A lower limit of average grey level difference has been defined : 20. The average grey level difference of the tissue paper sheet according to the invention is more than 20. It is believed that this characteristic is correlated with the smooth and translucent aspect of the simulated watermark disclosed therein.

**[0081]** With regard to the glossy appearance, or brightness of the simulated watermark pattern, it is linked to the reflection of light. The incident light rays are reflected on the marked pattern surface into one single direction whereas

- the same are reflected on the unmarked surface into a plurality of directions by the patches of the tissue paper sheet surface. The grey level values by image analysis with a lighting directed onto the sheet surface, show the differences between the marked regions which are black and the unmarked regions which are lighter and have a grey level more heterogenous resulting from the surface patches and the fluffy appearence.
- [0082] The brightness of the tissue paper has been measured by the means of a mini GLOSSMATER ERICHSEN (Model 507) apparatus. The measuring window is a rectangle the area of which is 13 cm<sup>2</sup>. The incident lighting has an angle of 60° out of the verticale. The weight per cent of reflected light is measured with regard to the surface state of the sample.

**[0083]** The values have been measured on black and white backgrounds. The brightness values of the tissue paper with a simulated watermark pattern according to the invention are generally higher than the ones of the tissue paper with a « press-marking » pattern illustrated by Figure 3, A.

- 30 with a « press-marking » pattern illustrated by Figure 3, A.
  [0084] Reffering to figure 2, the two plies 6 and 7 are advantageously bonded or connected together by the watermarked pattern 5'. The sheet generally comprises between one and three plies. When the sheet comprises several plies, the object of the invention is especially to bond the plies together. The quality and appearance of the watermark are directly related to the mutual bonding of the plies.
- **[0085]** A test is performed to analyze the behaviour of a tissue paper sheet with a simulated watermark pattern according to the figure 3C<sub>2</sub> (samples 1 and 2), comprising three plies, compared with a tissue paper sheet having a similar embossed pattern (T), comprising three plies. The embossing of the control sheet is an ordinary embossing without adhesive. Samples 1 and 2 and T having a width of 50 mm, have been cut.
- [0086] The delaminating test consists of measuring the average force of delaminating a sample at an angle of 180° and a constant speed of 100 mm/min. There is no value for the samples corresponding to the embossed pattern since the plies divide each other before measuring, in particular between the first two plies. With regard to the samples according to the invention, the average of the plotted values during the continue testing of delaminating is not representative. Only the pic values corresponding to the pattern regions when the plies are bonded, can be interpreted.
  [0087] The maximum value of forces to delaminate the plies are reported in the following table.
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	Force (Newton)					
	Facing th	ne engine	Cross wise			
	Between the 1st	Between the 2nd	Between the 1st	Between the 2nd		
······································	and 2nd plies	and 3rd plies	and 2nd plies	and 3rd plies		
T	-	0.008		0.07		
Sample 1	0.08	0.04	0.11	0.07		
Sample 2	0.05	0.02	0.15	0.11		

**[0088]** Even though these values are low, they show that the delamination of the samples according to the invention are higher than those of the embossed control sheet.

**[0089]** For a similar pattern, the simulated watermark tissue paper sheet according to the invention allows the plies to bond together contrarily to the embossed tissue paper sheet.

<sup>5</sup> **[0090]** The average thickness of the watermarked sheet has not changed. The sheet has kept all its physical and mechanical properties, the strength properties have not decreased and the elongation of the sheet has not been changed. In particular, the softness properties have not been adversely affected.

**[0091]** The main step in the process for marking with an impressed pattern resembling a watermark, a sheet of absorbent paper made of tissue paper, comprising one or more plies, is the deformation under stress of the sheet by at least one pass between a hard engraved roll and a receiving roll having a hardness of more than Shore-D 40, the

10 at least one pass between a hard engraved roll and a receiving roll having a hardness of more than Shore-D 40, the specific pressure exerted on the marked or watermarked simulating surface being at least 14,7 Mpa (150 kg/cm<sup>2</sup>) and the linear marking speed being at least 50 m/min.

[0092] Preferably, the sheet makes at least two passes between an engraved roll and a receiving roll.

- [0093] The resulting watermark may be distinctly improved by playing with the following parameters.
- <sup>15</sup> **[0094]** The nature and hardness of the receiving roll are essential for marking quality. Preferably, receiving rolls having a hardness greater than Shore-D 80 are used.

**[0095]** Heating the engraved roll also considerably improves the marking. The heating temperature lies within the interval ranging from approximately  $50^{\circ}$ C to approximately  $100^{\circ}$ C and preferably from approximately  $75^{\circ}$ C to approximately  $85^{\circ}$ C.

<sup>20</sup> **[0096]** Supplying the sheet, before marking, with water in liquid or steam form has a surprising effect on the quality of the simulated watermark.

**[0097]** These parameters are described in more detail in the description which follows with reference to Figure 4, which illustrates an embodiment of the process according to the invention.

[0098] Before the sheet undergoes the process, it initially comprises one ore more superimposed and non-connected plies, each stemming from a conventional process for manufacturing absorbent tissue paper and being dried. The sheet may or may not be initially creped and/or calendered. The sheet may also be impregnated with a softening lotion partly or essentially comprising fatty components.

**[0099]** The process for marking according to the invention can be done on the papermachine calender when the drying of the sheet is completed.

<sup>30</sup> **[0100]** The process according to the invention is preferably entirely separate from the process for manufacturing the sheet. This is a conversion process closely related to simulated watermarking processes, which may be placed in a unit other than that for manufacturing the sheet.

**[0101]** According to Figure 4, the sheet coming from the unwind roll 8 is brought into contact with water. Preferably, water is applied to the sheet in the form of steam by passing the sheet over a tank 9 containing water heated to a

- temperature greater than or equal to the boiling point of water. Steam 10 released from the tank comes into contact with the sheet surface which will subsequently undergo the deformation. Steam can also be applied with a steam pipe and a nozzle. The amount of steam applied is less than approximately 3 per cent by weight of the weight of the sheet and is preferably less than 2 per cent by weight. This supply of a small amount of water in the form of steam is a preparatory step for the next step in the formation of the watermark, considerably improving the quality of the watermark.
- <sup>40</sup> The steam has an effect on the definition and uniformity of the mark or simulated watermark. It is conceivable that the steam partially penetrates the sheet and acts on the papermarking fibers. Water may also be supplied in liquid form, for example by spraying fine droplets. The amount supplied is also less than 3, and preferably less than 2, per cent by weight of the weight of the sheet. Likewise, the definition of the simulated watermark is improved by supplying water in liquid form.
- <sup>45</sup> **[0102]** The next step is the formation proper of the simulated watermark. The sheet 1 then makes a first pass between a steel engraved roll 11 which is hard and a first receiving roll 12, and then a second pass between this same engraved roll 11 and a second receiving roll 13.

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**[0103]** The engraved roll 11 is heated to a temperature which may lie within the range of approximately  $50^{\circ}$ C to  $100^{\circ}$ C. Preferably, the heating temperature of this roll is  $75^{\circ}$ C. It has been observed that increasing the temperature up to  $75^{\circ}$ C has an influence on the transparency of the marked surfaces in the region of the patterns.

**[0104]** The combination of the application of water in the form of steam and of the heating of the engraved roll gives the best watermarking results.

**[0105]** The receiving rolls 12 and 13 have a high hardness, greater than Shore-D 80 and preferably greater than Shore-D 90. They may be made of paper, of composite material, or of any other material having this hardness.

<sup>55</sup> **[0106]** When the receiving roll surface hardness is within the range of approximately 80 to approximately 90 Shore-D, the receiving roll is preferably substantially elastic and deformable under the pressure exerted by the engraved roll. This means that the receiving roll surface tends to take or takes again its original form further to the pressure exerted by the engraved roll.

**[0107]** The cylinder elasticity is defined herein by the ratio of the energy restored by the cylinder following deformation to the energy that was imparted to it for deformation. This ratio can be determined from hysteresis cycles entailed by consecutive tension and relaxation using a dynamometer or by applying fly-based impact to a test bar.

**[0108]** Receiving rolls the surface of which have a hardness more than 30 HRC (Rockwell hardness), can be generally used. As for an example, the ceramic material fullfills this hardness characteristic. The receiving rolls may also be made of steel for very high hardness. As a matter of fact, these receiving rolls are first elastic and then have a plastic behaviour when a pressure is applied on the surface thereof.

**[0109]** In the case where the receiving roll surfaces have a hardness more than 30 HRC, the hard engraved roll, in particular a steel roll, can have a surface hardness which is higher or lower than the receiving roll surface hardness.

<sup>10</sup> **[0110]** The receiving cylinders may be of the same or different kind. Composite materials offer the best results in terms of resistance to mechanical and thermal stresses.

**[0111]** The specific pressure applied to the patterns or the local specific pressure is at least 14,7 MPa (150 kg/cm<sup>2</sup>). The force applied has been measured during the sheet's pass, under particular test conditions :

<sup>15</sup> - a pattern density or pattern distribution of 9 % ;

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- a roll configuration in which the steel engraved roll is arranged between a paper receiving roll, having a hardness of Shore-D 80 and a fixed central rotation axis, and a receiving roll having a hardness of Shore-D 92, on which the force is applied ; and
- an impression varying in this case from 4 mm to 8 mm (the impression arises from the deformation of the receiving
   roll by the engraved roll and corresponds to the width of the area of contact between them).

**[0112]** From the measured value of the force applied, the average specific pressure is calculated and then, from the latter and the surface area occupied by the engraved patterns, the local specific pressure on each pattern is calculated, this being at least 29,4 MPa (300 kg/cm<sup>2</sup>). Above a local specific pressure of approximately 29,4 MPa (300 kg/cm<sup>2</sup>), it has been found that the bonding of the plies and the definition of the watermark were improved.

[0113] The impression according to the pattern may be larger or smaller and tests have been carried out for impressions ranging from 4 to 12 millimetres, these values not being limiting.

**[0114]** The linear marking speed is at least 50 m/min. Maximum speeds of 300 m/min have been experimentally tested. However, under suitable conditions, higher theoretical speeds would be conceivable, in particular for new types

- <sup>30</sup> of cover for the receiving roll, making them more resistant to the stresses exerted. The transparency effect may be improved for a given speed by supplying steam or by heating the engraved roll. For example, the effect may be insufficient at a speed of 300 m/min with neither steam supply nor heating, whereas the effect is entirely satisfactory at this same speed with an engraved roll heated to 75°C. It has been found that the speed, heat and steam parameters act independently and may be cumulative.
- <sup>35</sup> **[0115]** The sheet may be calendered during the marking process or thereafter.

**[0116]** The process allows the engraving pattern to be easily changed by replacing the engraved roll in the sheetconversion installation according to the customer's choice.

**[0117]** A simulated watermark has been produced on a sheet of absorbent paper which had been impregnated beforehand with a softening lotion. It has been found that the fact of treating the surface of the sheet intending to be marked in no case changed the parameters of the marking process, or the actual simulated watermark on the finished product, whereas in certain processes in the prior art (for example the process described in US-A-4,481,243), the regions intended to be marked or embossed had not to be impregnated with a lotion. The properties of the lotion-impregnated sheet, which has been submitted to a simulated watermarking, were unchanged.

[0118] A device for implementing the process of the invention comprises at least one engraved roll, optionally heated, and at least one receiving roll.

[0119] One of these devices is illustrated by Figure 5.

**[0120]** This device comprises a steel roll 110 which is engraved and optionally heated. This roll 110 is arranged between two receiving rolls 120 and 130 which are substantially elastic further to the pressure exerted by the engraved roll 110. One of the rolls has a fixed central rotation axis and the other two rolls can move about an axis which is other

- 50 than their central rotation axis. In figure 4, the receiving roll 130 is fixed (its central rotation spindle 135 is fixed) and the roll 110 and 120 can move laterally. The roll 130 is fixedly mounted in the stack frame. The two rolls 110 and 120 are mounted in cases which are themselves positioned in vertical slideways. Stops are provided for the rolls. The upper mechanical stops can prove to be useful in the case of large vibrations. The lower stops serve to support the various rolls when the device is not running. The compressive force is exerted by the hydraulic rams 140 or by any equivalent
- means. The receiving roll 120 and the engraved roll 110 are applied, by means of pressure, against the receiving roll 130.
   [0121] Other devices are illustrated by figures 6 and 7.

**[0122]** According to figures 6 and 7, the central cylinder 200 or 300 is the engraved roll and is fixed. The other two rolls 201 and 202 or 301 and 302, are movable.

[0123] Compared with the rolls 110 and 120 of the device of figure 5, the rolls 201 and 202 of the device of figure 6 can come into contact with the fixed roll 200 by rotation.

[0124] In figure 7, the receiving rolls 301 and 302 come into contact with the fixed engraved roll 300 by a translatory movement.

5 [0125] The devices wherein the rolls, engraved and receiving rolls, having other configurations, for example being not aligned, are also included.

**[0126]** Two engraved rolls can be contemplated. These engraved rolls can have same or different patterns engraved in their surface. The two impressed patterns can be applied on the same side of the sheet or each pattern is applied on each side, respectively.

10 [0127] When the impression corresponding to each pattern is applied on the same side of the sheet and if the pattern of one engraved roll is different from the pattern of the other engraved roll, those patterns can preferably be complementary to obtain a complex pattern on the tissue paper sheet.

[0128] The present invention also encompasses tissue paper sheet comprising one or more plies and having on the one hand, patterns obtained by embossing or « press-marking », and, on the other hand, simulated watermark patterns obtained according to the process disclosed in the preceeding description. The embossing allows the sheet to increase its thickness, and the simulated watermarking allows the sheet to obtain an attractive aesthetic effect.

**[0129]** The simulated watermarking can be applied before or after the embossing or press-marking step.

#### 20 Claims

- An absorbent tissue paper sheet comprising one or more plies, each having a basis weight of approximately 12 1. to approximately 70 g/m<sup>2</sup> and a part of the surface of which is marked, characterized in that:
- 25 the said surface part is marked by an impressed pattern simulating a watermark, said impression being applied to one side of the sheet, the opposite side corresponding to said impression being plane,
  - said marked surface being smooth, glossy and translucent, and
  - said marked surface comprising at least one marked unit region wherein the ratio of the thickness of said marked unit region to the area of said marked unit region is less than 0.027 mm/mm<sup>2</sup>.
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- 2. A sheet according to claim 1, characterized in that with regard to the light transmission through said sheet, the difference between the average grey level of the marked surface and the average grey level of the unmarked surface is at least 20 by image analysis.
- 35 3. A sheet according to claim 1 or 2, characterized in that the ratio of the marked surface area to the total surface area of the sheet is less than 30 per cent.
  - A sheet according to one of the preceeding claims, characterized in that it comprises at least two plies joined 4. together by the applied pattern.
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- 5. A sheet according to one of the preceeding claims, characterized in that each ply has a basis weight of approximately 12 to approximately 50 g/m<sup>2</sup>.
- 6. A process for marking with an impressed pattern resembling a watermark a surface part of an absorbent tissue 45 paper sheet comprising one or more plies each having a basis weight of approximately 12 to 70 g/m<sup>2</sup>, the marked surface part comprising at least one marked unit region wherein the ratio of the thickness of said marked unit region to the area of said marked unit region is less than 0.027 mm/mm<sup>2</sup>, said process consisting in deforming, under stress, the said sheet by at least one pass between a hard engraved roll and a receiving roll having a surface hardness of more than Shore-D 40, the specific pressure exerted on the marked surface being at least 14,7 MPa (150 kg/cm<sup>2</sup>) and the linear marking speed being at least 50 m/min.
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- 7. A process according to claim 6, characterized in that the hardness of the receiving roll is at least Shore-D 80.
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- A process according to claim 7, characterized in that the hardness of the receiving roll is greater than Shore-D 90. 8.
- 9. A process according to one of claims 6 to 8, characterized in that the receiving roll is substantially elastic and yieldable under the pressure exerted by the engraved roll.

- **10.** A process according to claim 6, **characterized in that** the receiving roll has a surface hardness more than 30 HRC.
- **11.** A process according to one of claims 6 to 10, **characterized in that** the specific pressure exerted on the marked surface is at least 29,4 MPa (300 kg/cm<sup>2</sup>).
- **12.** A process according to one of claims 6 to 11, **characterized in that** the said engraved roll, preferably made of steel, is heated to a temperature ranging from approximately 50°C to approximately 100°C.
- **13.** A process according to claim 12, **characterized in that** the said engraved roll is heated to a temperature from approximately 75°C to approximately 85°C.
  - **14.** A process according to one of claims 6 to 13, **characterized in that** it comprises a preliminary step of treating the sheet before deformation, consisting in bringing the surface of the sheet into contact with a limited amount of water, in the form of liquid or steam, which is less than 3 per cent by weight with respect to the weight of the sheet.
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- **15.** A process according to claim 14, **characterized in that**, preferably, an amount less than 2 per cent by weight of steam is applied to the sheet.
- **16.** A process according to one of claims 6 to 15, **characterized in that** the sheet is marked by two passes between a hard and non-deformable engraved roll and a receiving roll whose surface is substantially elastic.
  - **17.** A process according to one claims 6 to 16, **characterized in that** the marking speed varies within the interval ranging from approximately 50 to approximately 300 m/min.
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#### Patentansprüche

- Bogen aus saugfähigem Seidenpapier mit einer Lage oder mehreren Lagen, von denen jede eine flächenbezogene Masse von annähernd 12 bis annähernd 70 g/m<sup>2</sup> aufweist, und bei denen ein Teil der Oberfläche markiert ist, dadurch gekennzeichnet, daß:
  - der Teil der Oberfläche mit einem eingeprägten wasserzeichenähnlichen Muster versehen ist, wobei die Einprägung auf die eine Seite des Bogens aufgebracht ist und die andere, der Einprägung entsprechende Seite eben ist,
  - die markierte Oberfläche glatt, glänzend und durchscheinend ist, und
  - die markierte Oberfläche mindestens eine markierte Bereichseinheit umfaßt,

wobei das Verhältnis der Dicke der markierten Bereichseinheit zu der Fläche der markierten Bereichseinheit weniger als 0,027 mm/mm<sup>2</sup> beträgt.

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- 2. Bogen nach Anspruch 1, dadurch gekennzeichnet, daß in bezug auf die Lichtdurchlässigkeit durch den Bogen hindurch der Unterschied zwischen der durchschnittlichen Graustufe der markierten Oberfläche und der durchschnittlichen Graustufe der unmarkierten Oberfläche bei der Bildanalyse mindestens 20 beträgt.
- 45 **3.** Bogen nach Anspruch 1 oder 2, **dadurch gekennzeichnet**, **daß** das Verhältnis der markierten Oberfläche zu dem gesamten Oberflächenbereich des Bogens weniger als 30 Prozent beträgt.
  - 4. Bogen nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet**, **daß** dieser mindestens zwei durch das aufgebrachte Muster miteinander verbundene Lagen umfaßt.
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- **5.** Bogen nach einem der vorhergehenden Ansprüche, **dadurch gekennzeichnet**, **daß** jede Lage eine flächenbezogene Masse von annähernd 12 bis annähernd 50 g/m<sup>2</sup> aufweist.
- 6. Verfahren zum Versehen eines Oberflächenteils eines Bogens aus saugfähigem Seidenpapier mit einer Lage oder mit mehreren Lagen, von denen jede eine flächenbezogene Masse von annähernd 12 bis 70 g/m<sup>2</sup> aufweist, mit einem eingeprägten wasserzeichenähnlichen Muster, wobei der markierte Oberflächenteil mindestens eine markierte Bereichseinheit umfaßt, wobei das Verhältnis der Dicke der markierten Bereichseinheit zu der Fläche der markierten Bereichseinheit weniger als 0,027 mm/mm<sup>2</sup> beträgt, wobei das Verfahren darin besteht, den Bogen

unter Spannung durch mindestens einen Hindurchlauf zwischen einer harten Musterwalze und einer Aufnahmewalze mit einer Oberflächenhärte von mehr als Shore D 40 zu verformen, wobei der auf die markierte Fläche ausgeübte spezifische Druck mindestens 14,7 MPa (150 kg/cm<sup>2</sup>) beträgt und die lineare Markierungsgeschwindigkeit mindestens 50 m/min beträgt.

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- 7. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß die Härte der Aufnahmewalze mindestens Shore D 80 beträgt.
- Verfahren nach Anspruch 7, dadurch gekennzeichnet, daß die Härte der Aufnahmewalze mehr als Shore D 90 beträgt.
  - 9. Verfahren nach einem der Ansprüche 6 bis 8, dadurch gekennzeichnet, daß die Aufnahmewalze im wesentlichen elastisch und unter dem von der Musterwalze ausgeübten Druck nachgiebig ist.
- 10. Verfahren nach Anspruch 6, dadurch gekennzeichnet, daß die Aufnahmewalze eine Oberflächenhärte von mehr als 30 HRC aufweist.
  - **11.** Verfahren nach einem der Ansprüche 6 bis 10, **dadurch gekennzeichnet**, **daß** der auf die markierte Fläche ausgeübte Druck mindestens 29,4 MPa (300 kg/cm<sup>2</sup>) beträgt.
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- **12.** Verfahren nach einem der Ansprüche 6 bis 11, **dadurch gekennzeichnet**, **daß** die vorzugsweise aus Stahl bestehende Musterwalze auf eine Temperatur im Bereich von annähernd 50 °C bis annähernd 100 °C erhitzt wird.
- **13.** Verfahren nach Anspruch 12, **dadurch gekennzeichnet**, **daß** die Musterwalze auf eine Temperatur im Bereich von annähernd 75 °C bis annähernd 85 °C erhitzt wird.
- 14. Verfahren nach einem der Ansprüche 6 bis 13, dadurch gekennzeichnet, daß dieses einen vorhergehenden Schritt zur Behandlung des Bogens vor dem Verformen umfaßt, der darin besteht, die Oberfläche des Bogens in Kontakt mit einer begrenzten Wassermenge in Form von Flüssigkeit oder Dampf zu bringen, die weniger als 3 Gew.-% beträgt, bezogen auf das Gewicht des Bogens.
- **15.** Verfahren nach Anspruch 14, **dadurch gekennzeichnet daß** vorzugsweise eine Menge von weniger als 2 Gew.-% Dampf auf den Bogen aufgebracht wird.
- 35 16. Verfahren nach einem der Ansprüche 6 bis 15, dadurch gekennzeichnet, daß der Bogen mit zwei Hindurchläufen zwischen einer harten und nicht verformbaren Musterwalze und einer Aufnahmewalze markiert wird, deren Ober-fläche im wesentlichen elastisch ist.
  - **17.** Verfahren nach einem der Ansprüche 6 bis 16, **dadurch gekennzeichnet**, **daß** sich die Markierungsgeschwindigkeit in einem Bereich ändert, der von annähernd 50 bis annähernd 300 m/min reicht.

#### Revendications

- Feuille de ouate de cellulose absorbant comprenant une ou plusieurs épaisseurs, ayant chacune un grammage d'environ 12 à environ 70 g/m<sup>2</sup> et dont une partie de la surface est marquée, caractérisée en ce que:
  - ladite partie de surface est marquée par un motif imprimé simulant un filigrane, ladite impression étant appliquée d'un côté de la feuille, le côté opposé correspondant à ladite impression étant plan,
  - ladite surface marquée étant lisse, brillante et translucide, et
  - ladite surface marquée comprenant au moins une région marquée unitaire dans laquelle le rapport de l'épaisseur de ladite région marquée unitaire à l'aire de ladite région marquée unitaire est inférieur à 0,027 mm/mm<sup>2</sup>.
  - Feuille selon la revendication 1, caractérisée en ce que concernant la transmission de lumière à travers ladite feuille, la différence entre le niveau de gris moyen de la surface marquée et le niveau de gris moyen de la surface non marquée est d'au moins 20, mesuré par analyse d'image.
    - 3. Feuille selon la revendication 1 ou 2, caractérisée en ce que le rapport de l'aire de surface marquée à l'aire de

la surface totale de la feuille est inférieur à 30%.

- **4.** Feuille selon l'une quelconque des revendications précédentes, **caractérisée en ce qu'**elle comprend au moins deux épaisseurs jointes l'une à l'autre par le motif appliqué.
- Feuille selon l'une quelconque des revendications précédentes, caractérisée en ce que chaque épaisseur a un grammage d'environ 12 à environ 50 g/m<sup>2</sup>.
- 6. Procédé pour marquer d'un motif imprimé ressemblant à un filigrane une partie de la surface d'une feuille de ouate de cellulose absorbant comprenant une ou plusieurs épaisseurs ayant chacune un grammage d'environ 12 à 70 g/m<sup>2</sup>, la partie de surface marquée comprenant au moins une région marquée unitaire dans laquelle le rapport de l'épaisseur de ladite région marquée unitaire à l'aire de ladite région marquée unitaire est inférieur à 0,027 mm/ mm<sup>2</sup>, ledit procédé consistant à déformer, sous contrainte, ladite feuille par au moins un passage entre un rouleau gravé dur et un rouleau récepteur ayant une dureté de surface supérieure à 40 Shore-D, la pression spécifique exercée sur la surface marquée étant d'au moins 14,7 MPa (150 kg/cm<sup>2</sup>) et la vitesse de marquage linéaire étant d'au moins 50 m/min.
  - 7. Procédé selon la revendication 6, caractérisé en ce que la dureté du rouleau récepteur est d'au moins 80 Shore-D.
- Procédé selon la revendication 7, caractérisé en ce que la dureté du rouleau récepteur est supérieure à 90 Shore D.
  - 9. Procédé selon l'une quelconque des revendications 6 à 8, caractérisé en ce que le rouleau récepteur est sensiblement élastique et déformable par la pression exercée par le rouleau gravé.
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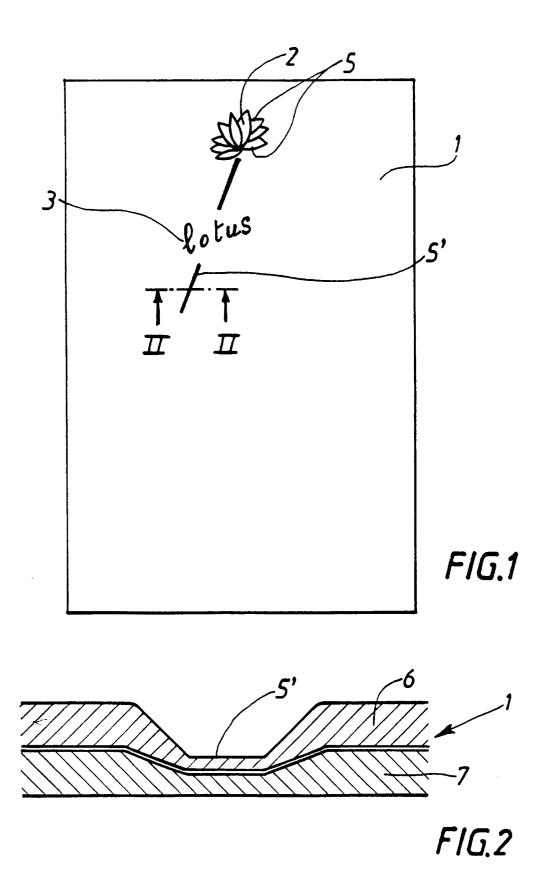
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- **10.** Procédé selon la revendication 6, **caractérisé en ce que** le rouleau récepteur a une dureté de surface supérieure à 30 HRC.
- Procédé selon l'une quelconque des revendications 6 à 10, caractérisé en ce que la pression spécifique exercée sur la surface marquée est d'au moins 29,4 MPa (300 kg/cm<sup>2</sup>).
  - **12.** Procédé selon l'une quelconque des revendications 6 à 11, **caractérisé en ce que** ledit rouleau gravé, de préférence fait d'acier, est chauffé à une température allant d'environ 50°C à environ 100°C.
- <sup>35</sup> 13. Procédé selon la revendication 12, caractérisé en ce que ledit rouleau gravé est chauffé à une température allant d'environ 75°C à environ 85°C.
  - 14. Procédé selon l'une quelconque des revendications 6 à 13, caractérisé en ce qu'il comprend en outre une étape préliminaire de traitement de la feuille avant déformation, consistant à amener la surface de la feuille en contact avec une quantité limitée d'eau, sous la forme de liquide ou de vapeur, qui est inférieure à 3% en poids par rapport au poids de la feuille.
  - **15.** Procédé selon la revendication 14, **caractérisé en ce que**, de préférence, une quantité de vapeur inférieure à 2% en poids est appliquée sur la feuille.
- 45

40

- 16. Procédé selon l'une quelconque des revendications 6 à 15, caractérisé en ce que la feuille est marquée par deux passages entre un rouleau gravé dur et indéformable et un rouleau récepteur dont la surface est sensiblement élastique.
- 50 17. Procédé selon l'une quelconque des revendications 6 à 16, caractérisé en ce que la vitesse de marquage varie dans un intervalle d'environ 50 à environ 300 m/min.



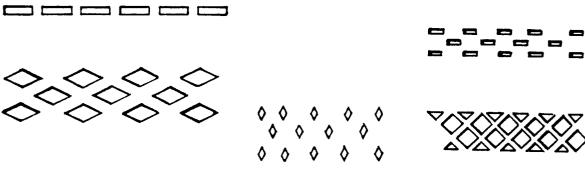
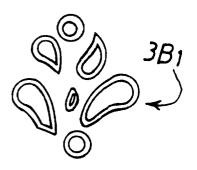


FIG.3A



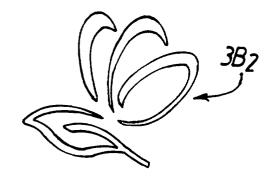
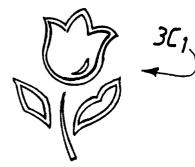


FIG. 3B



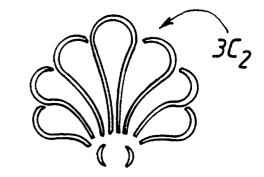
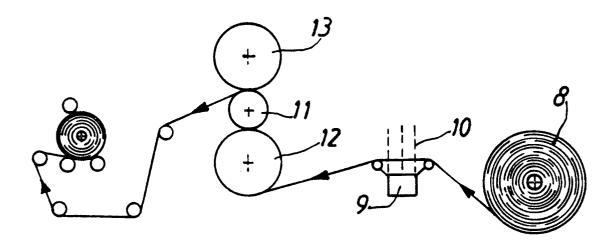
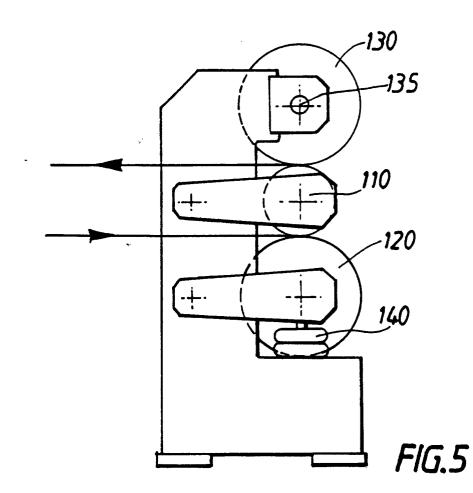


FIG.3C



*FIG.* 4



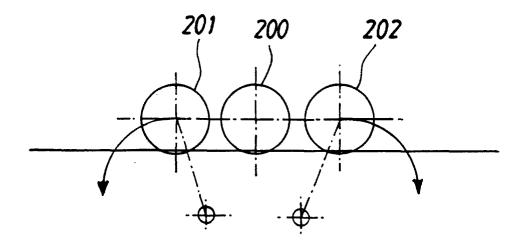
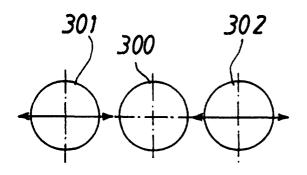


FIG.6



*FIG.7*