

(19)



Europäisches Patentamt  
European Patent Office  
Office européen des brevets

(11) Publication number:

**0 360 383**  
**A2**

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number: 89306807.2

(51) Int. Cl.<sup>5</sup>: D06M 15/423 , D06M 15/643

(22) Date of filing: 04.07.89

(30) Priority: 21.09.88 US 247199

(43) Date of publication of application:  
28.03.90 Bulletin 90/13

(84) Designated Contracting States:  
BE DE ES FR GB GR IT NL

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(54) Resilient cotton fiber and related method.

(57) An improved fiber product comprises individual fibers which are finish-treated by saturating the fibers in a finish solution of durable press resin and a lubricant, then drying and curing the fibers such that the resin is saturated and cured and cross-linked to the interior of the fibers while the lubricant is maintained at or near the surface of the fibers. A preferred method includes treating bleached cotton fibers with a finish solution containing 3.0% - 4.0% of a formaldehyde-free, alkylated glyoxyl/cyclic urea condensate resin and 1.5% -2.5% of a reactive silicone, and a catalyst. The resultant finish-treated cotton fibers have a resiliency greater than untreated fibers and comparable to or greater than polyester fibers.

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## RESILIENT COTTON FIBER AND RELATED METHOD

Field of The Invention

This invention generally relates to a process of treating individual fibers to have an improved resiliency, and more particularly, to treating individual fibers with a finish solution containing a durable press resin and a lubricant so that the fibers have improved resiliency and other properties suitable as a fiber fill material and for other uses.

Background Art

It is well known in fabric art to treat fabric made of cellulose fibers with urea-formaldehyde resins and other additives, including organopolysiloxanes, to provide wrinkle-resistant (durable press) properties. In particular, U.S. Patent 4,170,581 of Griffin, 3,177,093 of van Loo, and 2,758,946 of Spalding together are typical of the prior art teaching durable press fabric treatments using formaldehyde-based glyoxyl resin as a crosslinking agent to give durable press properties, and organopolysiloxanes as a soil release or water repellent agent. To overcome the problem of release of free formaldehyde by durable press treated fabrics, U.S. Patent 4,269,603 of Worth teaches the use of a formaldehyde-free finishing agent containing glyoxyl with reactive silicone and a catalyst. Alternatively, U.S. Patent 4,345,063 of North teaches durable press treatment using alkylated condensate resin products of the reaction of glyoxyl and cyclic ureas which are free of formaldehyde.

In home furnishing products, it is desirable to use fiber fill materials which have good resiliency and can regain their original volume despite repeated use. Polyester fibers are normally used because of their resiliency. Although cotton fibers would be preferred as a fill material because of their lower cost and comfortableness of feel, they do not have the resiliency of polyester fibers, and tend to become clumpy or matted and do not retain their original volume after repeated use. Thus, cotton and other natural fibers have heretofore not been suitable as a fiber fill material for the home furnishing market. Moreover, for absorbent layers and laminates of various types, it is desirable to use fibers which can retain their original volume during use.

Summary of The Invention

It is therefore principal object of the invention to provide a process for treating individual fibers to have an improved resiliency. In particular, the invention seeks to provide a treatment for cellulosic fibers, such as cotton fibers, to have improved properties suitable for a fiber fill material. A further object is to provide an improved fiber product of individual fibers which are finish-treated to have an improved resiliency and can maintain their volume.

In accordance with the invention, a process for treating individual fibers to have an improved resiliency comprises the steps of saturating the individual fibers with a solution containing a durable press resin and a lubricant such that the resin saturates to the interior of the fibers while the lubricant is retained at or near the surface of the fibers, and drying and curing the resin to be cross-linked to the fibers before the lubricant can be absorbed into the interior of the fibers. The lubricant is selected to have a larger molecular structure than the resin, so that the resin is absorbed more readily into the interior of the fibers than the lubricant.

In a preferred method of the invention, scoured and bleached cotton fibers are saturated with a solution containing a formaldehyde-free, alkylated glyoxyl/cyclic urea condensate resin and an amino-group silicone, then dried and cured. The resultant fibers have a resiliency equal to or better than polyester fiber. The resin component gives the treated fibers resiliency, while the silicone imparts lubrication to the fibers to reduce interfiber friction to allow them to slip easily across each other.

As another aspect of the invention, an improved fiber product comprises individual fibers, and particularly individual cotton fibers, provided with a finish of a durable press resin and a lubricant, wherein the resin is saturated and cured and cross-linked to the interior of the fibers while the lubricant is retained at or near the surface of the fibers. The finish-treated cotton fibers have a resiliency of about 4.1 - 5.8 centimeters or more of recovery after initial compression compared to about 4.1 centimeters for polyester fibers, as measured by a compression-regain method of testing resiliency.

Other objects, features, and advantages of the present invention will become apparent from the

following detailed description of the best mode of practising the invention when considered with reference to the drawings, as follows:

## 5 Brief Description of The Drawings

Fig. 1A is a schematic diagram of a continuous process for treating individual fibers with a resiliency-improving finish in accordance with the invention;

Fig. 1B is a schematic diagram of a batch process for treating individual fibers with a resiliency-improving finish;

Fig. 2 is a cross-sectional diagram of a finish-treated cotton fiber in accordance with the invention;

Fig. 3 is a schematic diagram illustrating a method of testing the resiliency of the finish-treated fibers; and

Fig. 4 is a chart comparing the resiliency of finish-treated cotton fibers to untreated cotton fibers and polyester fibers.

## Detailed Description of Preferred Embodiments

Referring to Figs. 1A and 1B, two alternative processes for making the high resiliency fibers in accordance with the invention are shown generally comprising the steps of scouring and bleaching raw and unfinished fibers, saturating them with a finish solution containing a durable press resin and a lubricant, then drying and curing the fibers to produce the high resiliency fiber product (sometimes referred to herein as "hi-loft" fibers). In the following description, the particular example of standard mature cotton fibers is used, although the invention can also be applied to other cellulosic fibers, such as cotton linters, as well as to other fibers which have, or are treated to have, a structure which can absorb and cross-link with durable press resin.

In the continuous process option of Fig. 1A, bales of grieger cotton fibers are opened and cleaned with step cleaners. A carded mat of cotton fibers is formed. The mat is saturated in a caustic and surfactant solution in the scouring step, and passed through a scour steamer at a suitable temperature and time required for removal of basic impurities such as wax, oils, pectins, etc. The mat is saturated in a bleaching solution, such as hydrogen peroxide or sodium hypochlorite, to bleach the fibers white, passed through a bleach steamer at a suitable temperature and time required for bleaching to occur, and rinsed to remove the bleach liquor and adjust the pH of the fiber surface residues.

The scoured and bleached cotton fibers are then immersed in a finish bath containing the durable press resin, the lubricant, and a catalyst. The fibers are immersed such that the resin can saturate to the interior of the fibers while the lubricant remains at or near the surface of the fibers. The finish-treated fibers are then dried and cured within a time period before the lubricant can be absorbed into the interior of the fibers. The dried mat is then broken up in a tufter and baled as the hi-loft cotton fiber product.

The continuous process depends on the use of conveyors and other aids to transport the carded mat of fibers through the process. The mat is preferably in the range of 24 oz./sq.yd. The heavier weight helps maintain the integrity of the fibers through the continuous process. One advantage of this process is the locking of the fibers in an open web state before scouring and bleaching, in order to prevent the formation of ropes, strings, and neps associated with cotton fibers processed in batches.

In the kier process option of Fig. 1B, bales of grieger cotton fibers are opened and cleaned with step cleaners. The fibers are then saturated with a caustic and surfactant solution, and formed into large (for example, 600 lb.) cakes by placing the wet fibers into a rotating rub and mechanically stamping the wet mixture to make the cakes as uniform as possible. The mixture is then pressed in a hydraulic press to form a compressed cake of fibers having the appearance of a large doughnut.

The doughnut shaped cakes are stacked in a cage and placed in a kier where they are subjected to a standard scouring and bleaching step. The cakes are then removed from the kier, and each cake is dewatered, for example, by placing it in a hydroextractor and spinning to remove as much water as possible. The dewatered cakes are put in a cake attacker or wet picker and broken up into small wet tufts. At this stage, the finish solution containing the durable press resin, lubricant, and catalyst is applied to the fibers by spraying or immersion, and the fibers are then fed into a drier where they are dried and cured. Alternatively, the dewatered cakes can be directly immersed in a finish bath, then separated and formed in a mat for drying and curing. The fibers can be further processed through an opener to fluff them into individual fibers as much as possible, then baled and wrapped for shipment.

An important factor to achieving the improved fiber resiliency in the invention is that the resin saturates

and is cured and crosslinked to the interior of the fibers while the lubricant stays at or near the surface of the fibers. As depicted in Fig. 2, a cotton fiber has an outer sheath and an interior core (lumen). The cotton fibers are scoured and bleached to remove their natural surface repellancy, and to render them absorbent so that they will quickly absorb the resin into the interior of the fibers. The lubricant will also tend to be absorbed into the fibers. Therefore, the resin is selected to have a smaller molecular structure than the lubricant, so that it is more readily absorbed into the interior while the lubricant stays on the surface of the fibers. If the fibers are dried and allowed to sit for an extended time prior to curing, for example, more than seven or eight hours, the lubricant may be gradually absorbed into the interior of the fibers. This would reduce the desired resilient properties of the fibers. Moreover, the lubrication of the fibers would be reduced. Therefore, the curing of the fibers must follow the drying of the fibers within a relatively short period of time.

The finish solution applied to the fibers in the above-described processes preferably contains 1.0% - 4.0% formaldehyde-free alkylated glyoxyl/cyclic urea condensate resin, with 2.5% - 4.0% being the most preferred range, 1.5% - 2.5% reactive silicone, and 0.6% - 0.7% magnesium chloride hexahydrate as the catalyst in water as the carrying medium. Formaldehyde-free alkylated glyoxyl/cyclic urea condensate resins are obtained, as disclosed in U.S. Patent 4,345,063 of North, by reacting cyclic urea and glyoxyl in the presence of a catalyst to yield a water-soluble oligomer condensate. The condensate is then wholly or partly alkylated by reacting it with an alcohol. Alternatively, glyoxal is reacted with an alkylated cyclic urea. The reaction product is a formaldehyde-free resin suitable for application as a finish to cellulose fibers as described above. Other resins which can be used include melamine resin, as well as formaldehyde-containing resins, such as glyoxyl (DMDHEU) resin. However, the latter are not as desirable for home furnishing or skin-contacting products because they can release formaldehyde. The resin should also be selected so that it does not bond the fibers together.

The lubricant is selected to have a lubricity which can overcome the interfiber surface friction, as well as a larger molecular structure than the resin. A reactive silicone can suitably be used, particularly an amino-functional group silicone, since it has an affinity for and tends to readily cover the hairy, scaly points on the surface of the cotton fibers. Other lubricants which can be used include stearates, such as butoxyethylstearate, or fatty acids. The catalyst is provided to promote cross-linking of the resin to the fibers. Preferred catalysts include metal salts, such as magnesium chloride hexahydrate.

The drying step can be carried out with dry cans providing radiant heat, or with forced hot air or an oven. The fibers are dried to 5% or less moisture before starting curing. Curing can be carried out from about 280 degrees to about 360 degrees Fahrenheit, and the curing time can vary from about 30 to 60 seconds depending on temperature. The preferred curing temperature is about 300 to 320 degrees. A curing temperature above 360 degrees may cause browning or yellowing of the silicone or resin finish on the cotton fibers.

The resultant finish-treated fibers are found to have an improved resiliency as measured by the compression-regain method of resiliency measurement. Referring to Fig. 3, the measurement is taken by compressing a sample of fibers in a cylinder with a plunger. For the comparative tests herein a one gram fiber sample is formed into a sliver and inserted into a standard 3/4 inch test tube. With the tube in a vertical position, a plunger with a one-pound weight attached is inserted in the tube and compresses the fiber sample under gravity. After a waiting period (5 minutes), the compressed height of the fibers is measured, and the plunger is removed. Following a recovery period (5 minutes), the recovery height of the fibers is measured. The difference between the compressed height and the recovery height is taken as a measurement of fiber resiliency.

Examples of cotton fibers treated with a range of finish formulations in accordance with the invention are shown in Table I below. The alkylated glyoxyl/cyclic urea condensate resin used was ZF-4, as sold by Sequa Chemicals of Chester, South Carolina. The silicone used was Sequa Soft-89 silicone, and the catalyst was #531, 30% magnesium chloride hexahydrate solution, also sold by Sequa Chemicals. As comparative examples, cotton fibers treated only with a surfactant (S634 cationic soap from Sequa Chemicals) and polyester fibers (PET) were also measured for resiliency.

TABLE I

	#1	#2	#3	#4	#5	#6
Fibers:	Cotton	Cotton	Cotton	Cotton	Cotton	PET
Resin:	3.0%	4.0%	3.0%		3.0%	
Silicone:	1.5%	2.0%	2.5%		1.5%	
Catalyst:	0.6%	0.7%	0.6%		0.6%	
Surfactant:				0.2%		

As shown in the graph of Fig. 4, the treated cotton fibers of Table I had a resiliency measured as 4.1 - 5.8 cm. or more of recovery, compared to 2.4 cm. for untreated (surfactant only) cotton fibers and 4.1 cm. for polyester fibers. Thus, the resin/silicone finish treatment provided a superior resiliency to the cotton fibers comparable to or substantially greater than that of polyester fibers. Other tests of treated cotton fiber using lesser amounts of resin than 3.0% also showed an improved resiliency over unfinished cotton fibers, though not as high as polyester fibers. Similar results can be obtained for other types of cellulosic fibers, such as cotton linters, and other fibers.

The durable press resin/lubricant treated fibers of the invention have the properties of increased resiliency as well as lubrication to allow them to slide easily over each other to maximize the resiliency effect. Treatment with resin alone will impart lesser resiliency properties to the cotton fibers, in the range of one-third to one-half that of the cotton fibers treated with resin and lubricant. The bleached cotton fibers tended to have 100% pickup of resin and lubricant from the finish solution. The resulting treated fibers therefore preferably have about 0.675 - 1.8 grams of resin per 100 grams of fiber. The preferred range of silicone was about 0.13 - 1.125 grams to 100 grams of fiber. The use of formaldehyde-free alkylated glyoxyl/cyclic urea condensate resin also resulted in almost a complete absence of formaldehyde from the finish, i.e. below trace amounts. The absence of formaldehyde permits the treated fibers to be safely used as fiber fill material for home furnishing products and other uses which involve skin contact.

The increased resiliency of the finish-treated fibers further results in an enhanced softness and an increased void volume being maintained around the fibers. Thus, the treated fibers can be advantageously used to form soft, hi-loft fill layers in laminates and fabrics. The resilient fibers can also be used to form highly absorbent layers due to their maintenance of the void volume around the fibers and the capacity to absorb and retain moisture in the void volume. For example, the resilient fibers can be mixed with absorbent fibers, such as wood pulp fibers, superabsorbent polymer particles or other absorbent materials. The resilient fibers maintain the void volume used by the absorbent materials to absorb moisture, thereby enhancing the absorptive capacity of the layer. If the fibers are treated with a hydrophilic lubricant, such as glycerine or butoxyethylstearate, the absorption capacity of the fibers is retained, thereby increasing the total absorptive capacity of the layer.

An absorbent layer structure using the resilient finish-treated fibers can have greater absorbency than a conventional superabsorbent layer using untreated cotton fibers and even synthetic fibers, due to the enhanced resiliency and lofted volume of the finish-treated fibers. Suitable applications for the absorbent layer structure include diaper absorbent sheets, sanitary napkins, absorbent sheets, tissues, towels, etc.

It is to be understood that although preferred embodiments of the invention have been described, numerous modifications and variations are of course possible within the principles of the invention. All such embodiments, modifications, and variations are considered to be within the spirit and scope of the invention as defined in the claims appended hereto.

## Claims

1. A method of treating individual fibers to have an improved resiliency, characterized by saturating the individual fibers with a finish solution of a durable press resin and a lubricant such that the resin saturates to the interior of the fibers while the lubricant is retained at or near the surface of the fibers, and drying and curing the resin to be cross-linked to the fibers before the lubricant can be absorbed into the interior of the fibers.

2. The method of claim 1, characterized by the fact that the lubricant is selected to have a larger molecular structure than that of the resin, so that the resin is absorbed more readily into the interior of the fibers than the lubricant.

3. The method of claim 1, characterized by the fact that the fibers to be finish-treated are scoured and bleached cotton fibers.

4. The method of claim 1, characterized by the fact that the durable press resin is an alkylated glyoxyl/cyclic urea condensate resin.

5 5. The method of claim 5 characterized by the fact that the resin is provided in an amount of 3.0% - 4.0% of the finish solution.

6. The method of claim 1, characterized by the fact that the lubricant is a amino-functional group silicone.

7. The method of claim 6, characterized by the fact that the silicone is provided in an amount of 1.5 - 2.5% of the finish solution.

8. The method of claim 1, characterized by the fact that the finish solution further includes a catalyst to promote cross-linking of the resin.

9. The method of claim 8, characterized by the fact that the catalyst is 30% magnesium chloride hexahydrate solution provided in an amount of 0.6% - 0.7% of the finish solution.

10 10. The method of claim 1 adapted for treating cotton fibers, characterized by the fact that it further comprises a continuous process of forming a carded mat of cotton fibers, scouring and bleaching the cotton fiber mat, then saturating the cotton fiber mat with the finish solution, then drying and curing the fibers.

11. The method of claim 1 adapted for treating cotton fibers, characterized by the fact that it comprises a batch process of forming compressed cakes of cotton fibers, scouring and bleaching the cotton fiber cakes in a kier, dewatering the cleaned cotton fiber cakes, separating the cotton fibers into small tufts, applying the finish solution to the cotton fibers, then drying and curing the fibers.

12. A finish-treated fiber product comprising individual fibers having a finish of a durable press resin and a lubricant, characterized by the fact that the resin is saturated and cured and cross-linked into the interior of the fibers while the lubricant is maintained at or near the surface of the fibers, such that the finish-treated fibers have an improved resiliency over untreated fibers.

13. The improved fiber product of claim 12, characterized by the fact that the lubricant is selected to have a larger molecular structure than that of the resin, so that the resin is absorbed more readily into the interior of the fibers than the lubricant.

14. The improved fiber product of claim 12, characterized by the fact that the fibers are cotton fibers.

15. The improved fiber product of claim 14, characterized by the fact that the resin is a formaldehyde-free alkylated glyoxyl/cyclic urea condensate resin.

16. The improved fiber product of claim 15, characterized by the fact tht the cotton fibers have a finish of 0.675 -1.8 grams of formaldehyde-free alkylated glyoxal/cyclic urea condensate resin per 100 grams of fiber, and about 0.13 - 1.125 grams of an amino-functional group silicone as the lubricant to 100 grams of fiber.

17. The improved fiber product of claim 15, characterized by the fact that the finish-treated fibers have a resiliency of about 4.1 - 5.8 centimeters or more of recovery after initial compression, using a compression-regain testing method, as compared to about 4.1 centimeters for polyester fibers.

18. The improved fiber product of claim 12, characterized by the fact that the finish-treated fibers are used as a fiber fill material.

19. The improved fiber product of claim 12, characterized by the fact that the finish-treated fibers are used to form an absorbent layer.

20. The improved fiber product of claim 12, characterized by the fact tht the lubricant is a hydrophilic lubricant.

## CONTINUOUS PROCESS OPTION

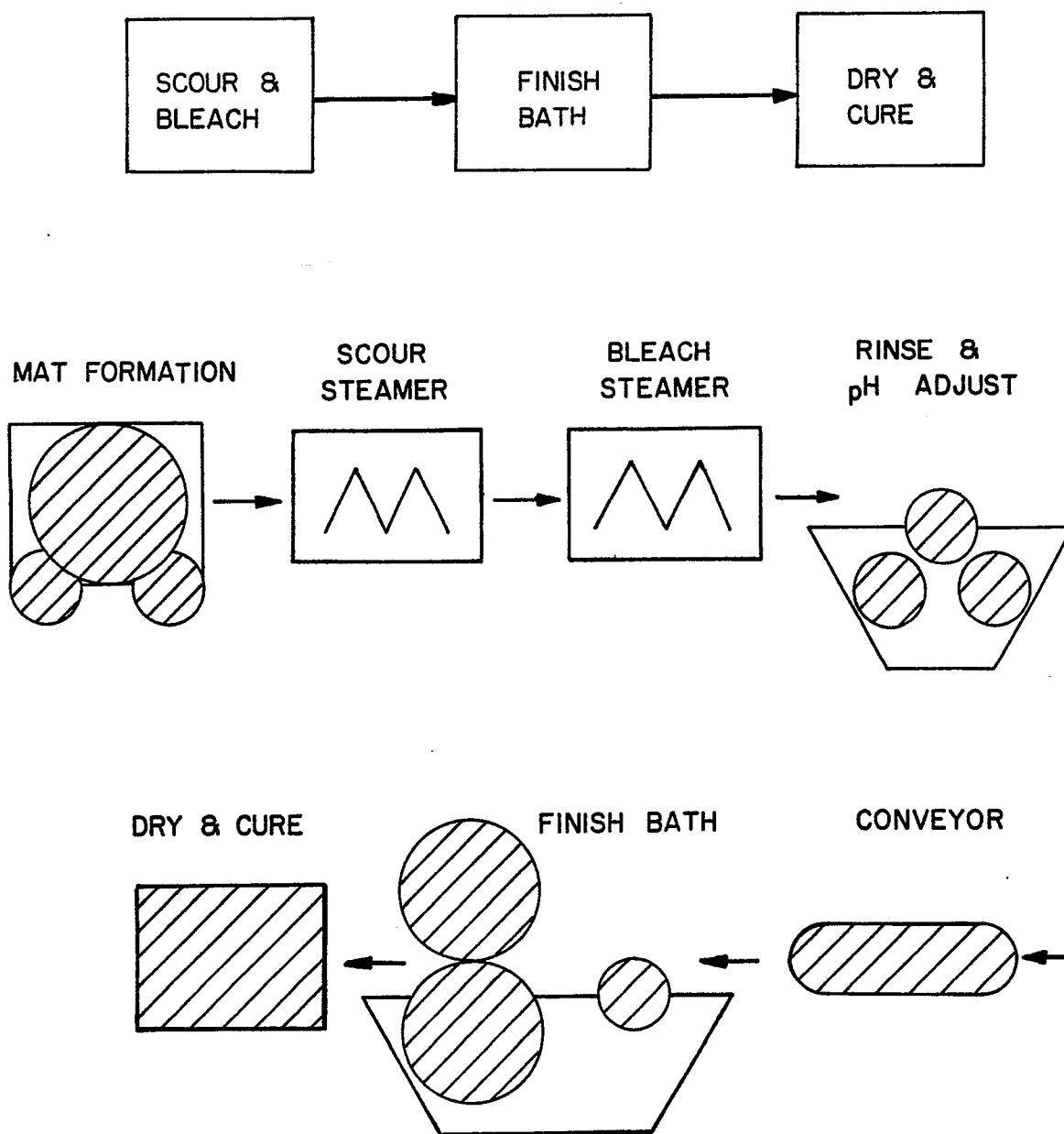


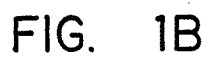
FIG. 1A

### KIER PROCESS OPTION

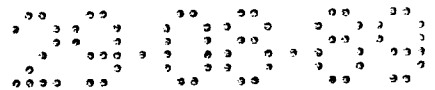
```
graph LR; A[SCOUR & BLEACH] --> B[FINISH BATH]; B --> C[DRY & CURE]
```

The Kier Process Option is a three-step sequential process:

- SCOUR & BLEACH
- FINISH BATH
- DRY & CURE







# COTTON FIBER

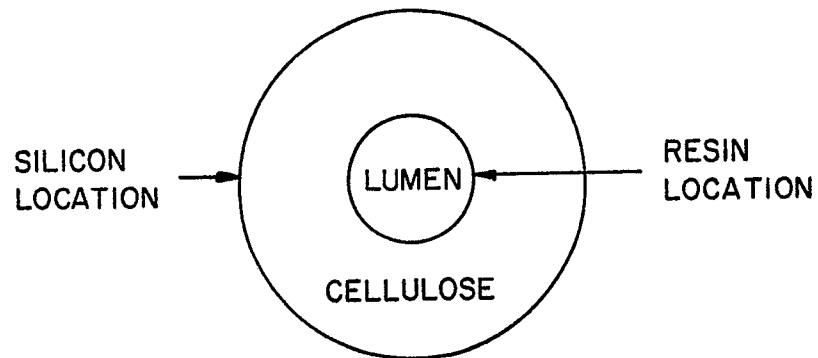


FIG. 2

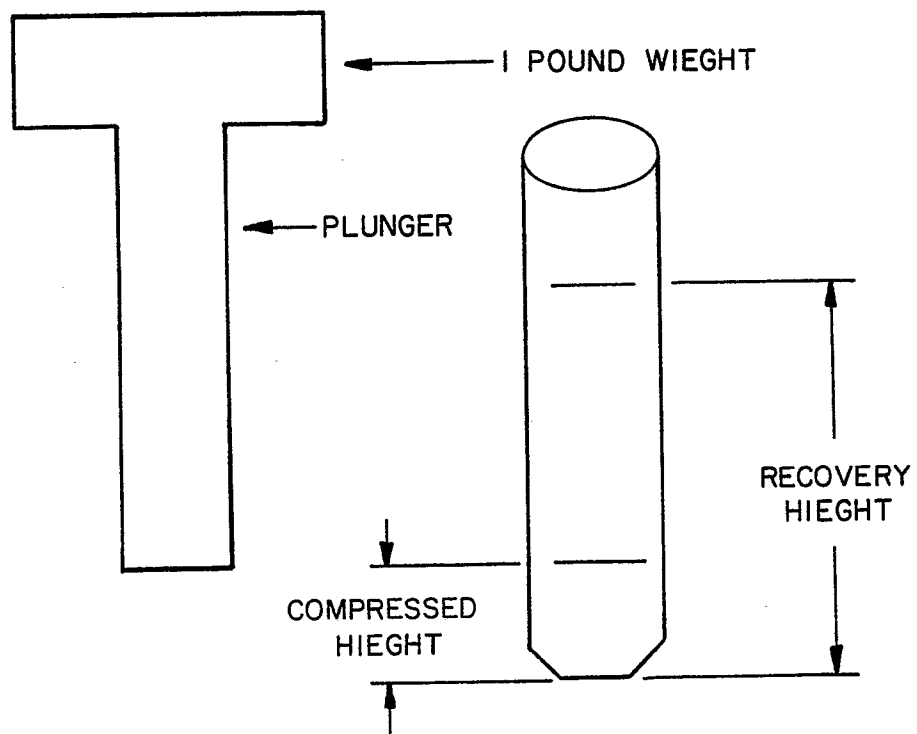
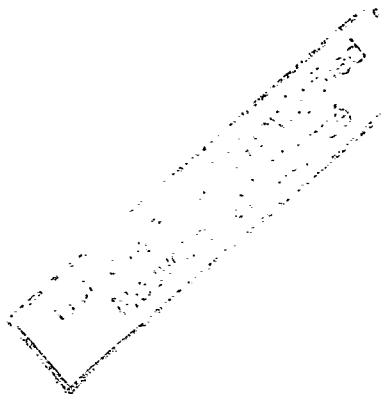
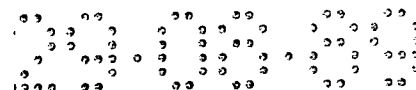


FIG. 3



## RESILIENCY TESTS

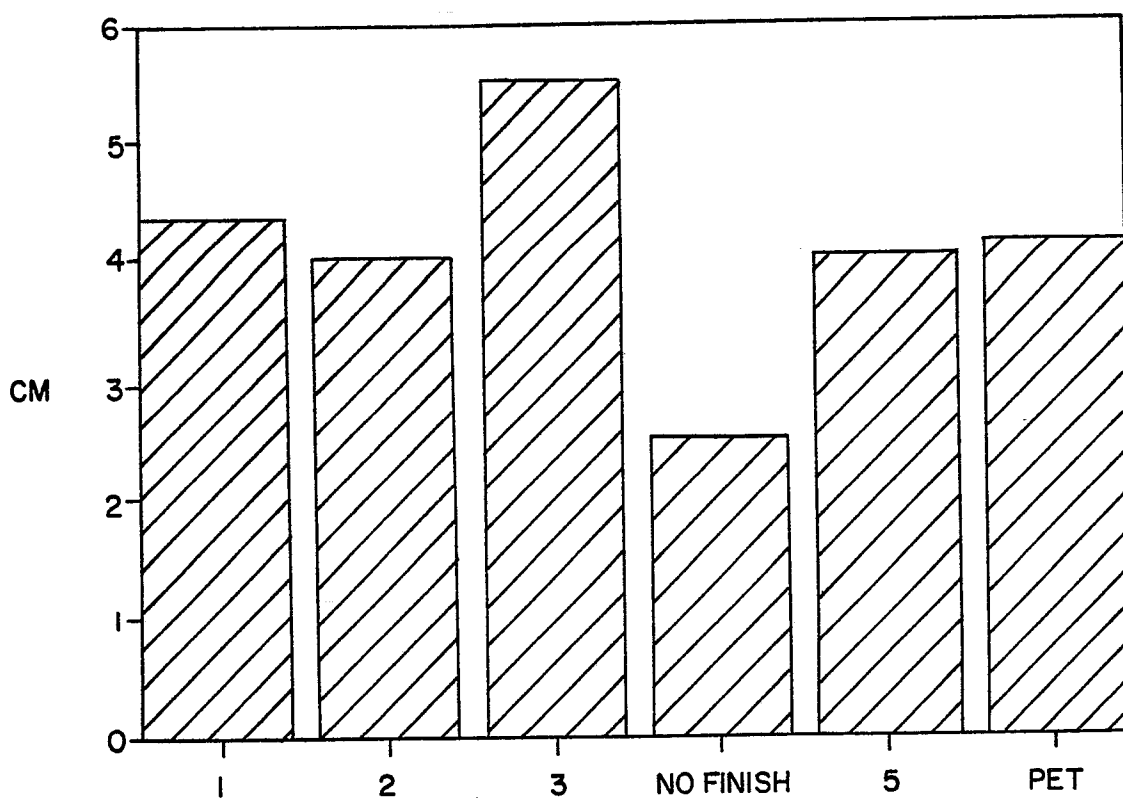


FIG. 4