



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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| <b>(54) Title:</b> THIOESTERS AS BOUNDARY LUBRICANTS<br><br><b>(57) Abstract</b><br><br>A textile fiber finish composition comprising: (a) from about 95 to about 99 % by weight of a lubricant component; and (b) from about 1 to about 5 % by weight of a boundary lubricant additive having the formula (I): $R_1-OOCCH_2CH_2-S-CH_2CH_2COO-R_2$ wherein $R_1$ and $R_2$ are each, independently, a $C_8-C_{18}$ alkyl group, all weights being based on the weight of the composition.                                                                                                                             |           |                                                                                                                                                                                                  |

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## THIOESTERS AS BOUNDARY LUBRICANTS

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### Field of the Invention:

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This invention relates to a composition and process for decreasing fiber-to-fiber and fiber-to-metal friction, as well as the scroop, in synthetic continuous filament fibers and scroop in staple fibers. More particularly, it has been surprisingly found that thioesters provide enhanced boundary lubrication in fiber finish compositions, thereby decreasing the fiber-to-fiber and fiber-to-metal friction coefficients, as well as scroop, in synthetic textile fibers.

### Background of the Invention:

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Finishing compositions are generally applied to textile fibers to improve their subsequent handling and processing. Fiber finishes play an important role in assisting the fiber producer to manufacture the product, and enable the fiber producer's customers to carry out the required yarn and fabric manufacturing processes to obtain the finished textile product. The formulation and amount of finish composition applied depend on a number of variables which include the chemical composition of the textile fiber, the particular stage in the processing of the fiber, and the end use under consideration.

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For example, compositions referred to as "spin finishes" are usually applied to textile fibers after extrusion. These or other finishes which may be applied to yarn prior to knitting or winding, and to fiber tows prior to or at the time of crimping, drying, cutting, drawing, roving, and spinning, or to staple fibers prior to carding, i.e., web formation, and subsequent textile operations such as yarn manufacture or preparation of nonwoven webs are commonly called secondary

or over-finishes. Such finishes provide lubrication, prevent static build-up, and afford a slight cohesion between adjacent fibers.

The application of such finishes is generally accomplished by contacting a fiber tow or yarn with a solution or an emulsion comprising at least one component having antistatic properties. In addition to a lubricant and anti-static agent, wetting agents, additives such as antioxidants, biocides, anti-corrosion agents, pH control agents, as well as emulsifiers are also commonly found in such finish mixtures. Finish compositions can also be applied to tow, yarn, or cut staple by spraying.

Acceptable finishes must fulfill a number of requirements in addition to providing desired lubricating and antistatic effects. For example, they should be easy to apply (and to remove if desired), they should have good thermal and chemical stability, they should not adversely affect the physical or chemical properties of the fibers to which they are applied and they should aid the subsequent processes to which the treated fibers are subjected, they should not leave residues on surfaces or cause toxic fumes or undesirable odors, they should provide for rapid wetting of fiber surfaces, they should be water-soluble or emulsifiable or solvent-soluble, they should have good storage stability, they should be compatible with sizes, nonwoven binders and other fiber treatments, they should not attract soil or cause color changes to the fibers, they should not interact with frictional elements used in texturizing and they should not be corrosive to machine parts.

The purpose of a fiber finish is to provide fiber to metal lubrication and fiber to fiber cohesion, as well as eliminate static electricity. Although much of the basic work to elucidate the mechanisms of lubrication was done in the distant past, results of this work continue to be used to understand and apply results of frictional testing to current problems and the development of new finishes.

The contribution of frictional and antistatic properties can be observed throughout fiber manufacturing and processing. An example is the case of a low denier polypropylene staple fiber which is to be carded into a web and thermally bonded for some disposable nonwoven application. This requires a formulation which in conjunction with the fiber crimp, contributes a relatively high fiber to fiber friction which is important in insuring a carded web with good cohesion, uniformity, and integrity, and which compensates for the low stiffness of the fibers. Low fiber to metal friction is also a key factor in the processing of these staple fibers which have diameters on the order of only 15 to 20 micrometers.

Another example involves a slit film or ribbon type yarn intended for woven carpet backing for tufted carpets. During its manufacture, good wetting of the fiber surface by the finish and moderate frictional coefficients are required. For tufting, however, relatively low fiber to metal friction is a very important feature because of the action of tufting needles on the backing fabric.

Finally, low fiber to fiber friction is a highly desirable feature of continuous filament yarns used in cordage applications which involve twisting and plying to form compact structures which have a large amount of fiber to fiber contact. Low friction is desirable since it is generally associated with high flex resistance, high

energy absorption and therefore, long life.

A different area of fiber-to-fiber friction is concerned with continuous filament yarns. This may be illustrated by some examples within the fiber manufacturing plant: package building in spinning and filament drawing or tow drawing are the major steps where the fiber-to-fiber friction is of critical importance. In yarn processing, yarn delivery in coning, stitch formation in knitting, filament damage in braiding, strength and elongation in cordage, slippage of weave in fabric, yarn-to-fabric friction in sewing, are some of the areas where yarn-to-yarn friction is important.

#### Summary of the Invention:

The present invention is directed to a textile fiber finish composition containing from about 95 to about 99% by weight of a lubricant component and from about 1 to about 5% by weight of a boundary lubricant additive having the formula I:



wherein  $R_1$  and  $R_2$  are each, independently, a  $C_8$ - $C_{18}$  alkyl group, all weights being based on the weight of the composition.

The present invention is also directed to a process for treating a textile fiber involving contacting the fiber with from about 0.3 to about 2.0% OWF (on-weight-of-fiber) of the above-disclosed textile lubricant composition.

#### Description of the Invention:

Other than in the operating examples, or where otherwise indicated, all numbers expressing quantities of ingredients or reaction conditions used herein

are to be understood as being modified in all instances by the term "about."

The two primary components of the textile fiber finish composition of the present invention are the lubricant component and the boundary lubricant additive.

5           The lubricant component of the fiber finish may be selected from the group consisting of  $C_{8-22}$  fatty acids, synthetic hydrocarbon oils, alkyl esters such as tridecyl stearate (EMEREST® 2308) which is the reaction product of tridecyl alcohol and stearic acid, polyols such as glycerin, and polyol esters such as trimethylol propane tripelargonate (EMERY® 6701) and pentaerythritol  
10           tetrapelargonate (EMERY® 2484).

          The lubricant component of this invention is emulsifiable and capable of forming a stable emulsion with water. By the term "stable emulsion" it is meant that the emulsion is stable at the time of application of the fiber finish composition onto the yarn surface. This is meant to include oil-in-water finishes  
15           which may be mixed just prior to their application to the yarn surface and which may be stable only under conditions of mixing and application. Typically, however, the finish will be mixed well prior to yarn application and then applied via various applicators from a storage tank or the like and thus the emulsion must be stable for extended time periods. It may also be desirable to include,  
20           as part of the lubricant component, an antistatic agent to aid in controlling the formation of static electricity during fiber processing. Thus, other components which may also be used as part of the lubricant of the present invention include emulsifiers and antistatic agents.

An emulsifier may be employed in the composition of the present invention, if required. The emulsifier may comprise any suitable emulsifying agent. Typical emulsifiers include an unethoxylated ester such as sorbitan monolaurate, an ethoxylated ester such as ethoxylated sorbitan monooleate, ethoxylated C<sub>8-22</sub> fatty acids such as the reaction product of ethylene oxide with pelargonic acid to form PEG 300 monopelargonate (EMEREST® 2634) and PEG 400 monopelargonate (EMEREST® 2654), the reaction product of ethylene oxide with coconut fatty acids to form PEG 400 monolaurate (cocoate) (EMEREST® 2650) and PEG 600 monolaurate (EMEREST® 2661), and the like, and an ethoxylated alcohol such as ethoxylated C<sub>11-C15</sub> alcohol or combination thereof. An alkali metal soap of a fatty acid such as potassium oleate may be included with an ethoxylate emulsifier, but it is not necessary. Preferred emulsifiers include an ethoxylated sorbitan monooleate (POE(5)) such as commercially available from Henkel Corporation, Mauldin, South Carolina, under the tradename EMSORB® 6901; POE (9) oleic acid under the tradename Emery 2646; and a polyethylene glycol ether of secondary alcohol commercially available under the tradename TERGITOL® 15-S-3 from Union Carbide Corporation, Danbury, CT.

An antistatic agent which may be employed generally comprises any suitable anionic, cationic, amphoteric or nonionic antistatic agent. Anionic antistatic agents are generally sulfates or phosphates such as the phosphate esters of alcohols or ethoxylated alcohols. Cationic antistatic agents are typified by the quaternary ammonium compounds and imidazolines which possess a

positive charge. Examples of nonionics include the polyoxyalkylene derivatives. The anionic and cationic materials tend to be more effective antistats.

As was mentioned above, if an emulsifier and/or an antistatic agent are used, they will comprise a part of the lubricant component, in addition to those lubricant component candidates already disclosed.

The boundary lubricant additive used in the present invention is a thioester having the formula I:



wherein  $R_1$  and  $R_2$  are each, independently, a  $C_8$ - $C_{18}$  alkyl group.

Examples of thioesters suitable for use as a boundary lubricant additive in the textile lubricant composition of the present invention include, but are not limited to, thiodipropionate derivatives such as di-2-ethylhexyl thiodipropionate, di-hexyldecyl thiodipropionate, di-lauryl thiodipropionate, and the like, commercially available from Henkel Corp. under the tradename STANDAPOL®.

A particularly preferred thioester for use in the present invention is di-isotridecyl thiodipropionate, commercially available from Cytec Corporation under the tradename CYANOX® 711.

According to one embodiment of the present invention, there is provided a textile lubricant composition containing from about 95 to about 99% by weight, and preferably from about 98 to about 99% by weight of a lubricant component, and from about 1 to about 5% by weight, and preferably from about 1 to about 2% by weight of the above-disclosed boundary lubricant additive, all weights being based on the weight of the textile lubricant composition.

In addition to the lubricant component and boundary lubricant additive, other textile finishing components may also be present in the textile fiber finish composition of the present invention. Examples thereof include, but are not limited to, wetting agents, biocides, anti-corrosion agents, and pH control agents.

5       The textile fiber finish composition of the present invention may be applied to virtually any type of fiber material including glass, cellulosics such as acetate, triacetate, rayon, non-cellulosics such as acrylics, modacrylic, nylon, aramid, olefins such as polyethylene and polypropylene, polybenzimidazole, polyesters such as polyethylene terephthalate and polybutylene terephthalate or  
10       copolyesters thereof, saran, spandex and vinyon.

According to another embodiment of the present invention, there is provided a process for lubricating a textile fiber in order to reduce fiber-to-fiber and fiber-to-metal friction coefficients, as well as scroop, involving contacting the fiber to be treated with the above-disclosed textile fiber finishing composition.

15       The process involves applying from about 0.3 to about 2.0% OWF ("on the weight of the fiber"), and preferably from about 0.5 to about 0.8% OWF of the textile fiber finish composition onto the fiber.

As was mentioned previously, the textile fiber finish composition will typically be applied onto the fibers to be treated in the form of an aqueous  
20       emulsion. Thus, the fibers will be contacted with the fiber finish composition at a percent actives concentration ranging from about 2 to about 20% actives, and preferably from about 8 to about 10% actives.

The present invention will be better understood from the examples which

follow, all of which are intended to be illustrative only and not meant to unduly limit the scope of the invention. Unless otherwise indicated, percentages are on a weight-by-weight basis.

Example I

5           A textile lubricant composition for fiber and textile applications was prepared having the following formulation.

|    | <u>Component</u>                                                               | <u>%/wt.</u> |
|----|--------------------------------------------------------------------------------|--------------|
|    | (a) diisotridecylthiodipropionate                                              | 1.0          |
|    | (b) coconut oil                                                                | 49.0         |
| 10 | (c) POE(16)hydrogenated castor oil                                             | 15.0         |
|    | (d) POE(5)C <sub>8-10</sub> /C <sub>16-18</sub> ether carboxylate methyl ester | 22.0         |
|    | (e) oleic diethanolamide                                                       | 8.0          |
|    | (f) sulfated castor oil, sodium salt                                           | <u>5.0</u>   |
|    |                                                                                | 100.0        |

15           The ingredients listed above were mixed together at ambient temperature using agitation to form a textile lubricant composition in accordance with the present invention. The lubricant composition was then applied onto nylon-6,6 fibers at 0.7% OWF.

A Comparative Example was also prepared having the following formulation:

|   | <u>Component</u>                                                               | <u>%/wt.</u> |
|---|--------------------------------------------------------------------------------|--------------|
| 5 | (a) coconut oil                                                                | 50.0         |
|   | (b) POE(16)hydrogenated castor oil                                             | 15.0         |
|   | (c) POE(5)C <sub>8-10</sub> /C <sub>16-18</sub> ether carboxylate methyl ester | 22.0         |
|   | (d) oleic diethanolamide                                                       | 8.0          |
|   | (e) sulfated castor oil, sodium salt                                           | <u>5.0</u>   |
|   |                                                                                | 100.0        |

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#### Lubricant Composition Evaluations

Frictional properties can be readily measured by applying known amounts of finish composition to yarns under controlled conditions in the laboratory. Recognizing that laboratory measurements at best only simulate actual use conditions, they have nevertheless been found to be a reasonably good predictor of behavior. One of the well-known instruments for performing frictional measurements is the Rothschild F Meter. In case of fiber to metal friction, the measurement is carried out by pulling a yarn around a circular metal pin under conditions of known pre-tension and angle of contact. The output tension is measured and the coefficient of friction determined from the capstan equation

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$$T_2/T_1 = e^{\mu \Theta}$$

where  $T_1$  and  $T_2$  are the incoming and outgoing tensions respectively,  $\Theta$  the

angle of contact in radians, and  $\mu$  the coefficient of friction. The Rothschild instrument calculates and plots the coefficient of friction automatically. Some prefer to use the value of  $T_2 - T_1$  as a measure of the frictional force since strictly speaking the capstan equation is not accurately obeyed by compressible materials such as fibers.

There are a number of variables, both mechanical and physical, in addition to the pretension and angle of contact, which can influence friction results. Some of these are speed, surface roughness, surface temperature, ambient temperature and humidity, finish composition viscosity, uniformity of finish composition application, finish composition concentration on the fiber, and fiber size and shape. Thus, when performing laboratory frictional experiments to determine the performance of a finish composition, one should select a condition related to that which the yarn will be exposed, such as for example, frictional measurements against a heated surface.

The fiber to fiber friction measurement is carried out in a similar way except that the yarn is twisted around itself and the force determined to pull the yarn in contact with itself. Again, with a knowledge of the incoming tension, the angle of wrap, and the outgoing tension, the frictional coefficient can be determined. In the case of fiber to fiber friction, it is customary to distinguish between static and dynamic frictional coefficients. Static friction is determined at a low speed (on the order of 1 cm/min), and dynamic friction at a higher speed. When measuring low speed friction, a stick-slip phenomenon is sometimes observed. It is this measurement which is most closely related

to the "scroop" observed with staple fibers, or the cohesion of staple fiber web as it emerges from a card, or the performance of a finish composition in yielding a yarn package which is stable and does not slough. The stick-slip phenomenon indicates that the static friction is higher than the dynamic friction and can be affected by the behavior of lubricants.

The lubricant composition of Example 1 containing a thioester boundary lubricant additive, and a comparative composition without the additive were applied onto nylon-6,6 fibers and tested to determine their effect on friction. The results thereof are found in the Tables below.

Table 1: Effect of Lubricant Composition on F/F Friction Based on Fiber

Pretension of 10 grams

| Example | 100<br>yards/min | 300<br>yards/min | 500<br>yards/min | Amount Applied<br>Onto Fibers |
|---------|------------------|------------------|------------------|-------------------------------|
| 1       | 56               | 72               | 78               | 0.7 OWF                       |
| Comp.   | 62               | 77               | 85               | 0.7 OWF                       |

As can be seen from the data in Table 1, by adding the boundary lubricant additive of the present invention to a textile lubricant composition, the fiber-fiber friction, measured as a function of pretension, is effectively lowered.

Table 2: Effect of Lubricant Composition on S/S at 0.002 cm/s

| Example | % OWF | Coefficient of Friction |
|---------|-------|-------------------------|
| 1       | 0.5   | 0.23                    |
| Comp.   | 0.5   | 0.25                    |
| 1       | 1.0   | 0.23                    |
| Comp.   | 1.0   | 0.24                    |

As can be seen from the data in Table 2, by adding the boundary lubricant additive of the present invention to a textile lubricant composition, the stick-slip is effectively lowered at a coefficient of friction of 0.002 cm/s.

Table 3: Effect of Lubricant Composition on S/S at 0.5 cm/min

| Example | F/F cN | Amplitude cN |
|---------|--------|--------------|
| 1       | 23.1   | 3.8          |
| Comp.   | 24.1   | 5.0          |

The lubricant composition may be applied onto the filament according to a variety of known procedures. For example, in the melt spinning process used for polypropylene manufacture, the polymer is melted and extruded through spinnerette holes into filaments which are cooled and solidified in an air stream or water bath. Shortly thereafter, they contact a lubricant composition applicator which can be in the form of a kiss roll rotating in a trough. The amount of active lubricant applied onto the filaments can be controlled by the concentration of lubricant composition in the solution or emulsion and the total wet pick-up. Alternatively, positive metering systems may be used which pump the lubricant

composition to a ceramic slot which allows the lubricant composition to contact the moving filaments.

From this point, the yarn which now has a coating of lubricant composition moves forward into any of several processes. The amount of lubricant composition to be applied onto a synthetic filament is also dependent on the end product of the filament yarn. If staple fiber is the desired product, the filament bundles are combined into large tows, oriented by stretching, crimped, and cut into short lengths for processing on textile equipment to ultimately make yarn or nonwoven webs. In this instance, it is the "scroop" of the fibers which is intended to be enhanced. In order to do so, it is preferred the lubricant composition have a concentration in the range of from about 0.5 to about 1.0% OWF, based on percent actives. If continuous filament yarn is the desired product, the filaments are also oriented but as discrete bundles containing a specific number of filaments and are wound as long continuous lengths.

In one version the unoriented or undrawn yarn is wound on a package, and drawn on a drawtwister. In another version called spin draw, the drawing operation is carried out in a continuous fashion on the same equipment without the step of winding the undrawn yarn.

Texturized yarns are also made as continuous filament yarns. Again, texturized yarns can be made by texturizing a fully oriented yarn or by simultaneously orienting and texturizing a partially oriented yarn.

In some of these processes the original textile lubricant composition application carries the fibers through the entire process. In others,

supplementary or overfinishes are applied somewhere later in the process.

The effect of frictional and static properties is generally obvious throughout fiber manufacture and processing. Fiber to fiber friction is important to the fiber producer in controlling formation and stability of filament yarn packages since sloughing can occur if it is too low. Also, if fiber to fiber friction is too low, there could be problems of poor web cohesion in carding of staple fibers. On the other hand, low fiber to fiber friction is very desirable for continuous filament yarns which are used in applications such as cordage which involves twisting and plying. Low friction is desirable since it is associated with high flex resistance and high energy absorption and therefore, long life. Fiber to metal friction is also very important in many of the fiber processes. Lower fiber to metal friction is generally preferred since there is less opportunity for damage to the fibers either by abrasion or heat generation as the yarn contacts metal surfaces.

What is claimed is:

1. A textile fiber finish composition comprising:

(a) from about 95 to about 99% by weight of a lubricant component; and

(b) from about 1 to about 5% by weight of a boundary lubricant additive

5 having the formula I:



wherein  $R_1$  and  $R_2$  are each, independently, a  $C_8$ - $C_{18}$  alkyl group, all weights being based on the weight of the composition.

2. The composition of claim 1 wherein the lubricant component is selected  
10 from the group consisting of  $C_{8-22}$  fatty acids, synthetic hydrocarbon oils, alkyl esters, polyols, polyol esters, emulsifiers, antistatic agents and mixtures thereof.

3. The composition of claim 1 wherein the lubricant component is present in the composition in an amount of from about 98 to about 99% by weight, based on the weight of the composition.

15 4. The composition of claim 1 wherein the boundary lubricant additive is a diisotridecylthiodipropionate.

5. The composition of claim 1 wherein the boundary lubricant additive is present in the composition in an amount of from about 1 to about 2% by weight, based on the weight of the composition.

20 6. The composition of claim 1 further comprising a component selected from the group consisting of a wetting agent, an antioxidant, a biocide, an anti-corrosion agent, a pH control agent, and mixtures thereof.

7. A process for lubricating textile fibers comprising contacting the textile fibers with a fiber finish composition comprising:

(a) from about 95 to about 99% by weight of a lubricant component; and

(b) from about 1 to about 5% by weight of a boundary lubricant additive

having the formula I:



wherein  $R_1$  and  $R_2$  are each, independently, a  $C_8$ - $C_{18}$  alkyl group, all weights being based on the weight of the composition.

8. The process of claim 7 wherein the fiber finish composition is in the form of an aqueous emulsion.

9. The process of claim 7 wherein the lubricant component is selected from the group consisting of  $C_{8-22}$  fatty acids, synthetic hydrocarbon oils, alkyl esters, polyols, polyol esters, emulsifiers, antistatic agents and mixtures thereof.

10. The process of claim 7 wherein the lubricant component is present in the composition in an amount of from about 98 to about 99% by weight, based on the weight of the composition.

11. The process of claim 7 wherein the boundary lubricant additive is a diisotridecylthiodipropionate.

12. The process of claim 7 wherein the boundary lubricant additive is present in the composition in an amount of from about 1 to about 2% by weight, based on the weight of the composition.

13. The process of claim 7 wherein the textile fibers are contacted with from about 0.3 to about 2.0% OWF of the textile lubricant composition.

14. The process of claim 8 wherein the fiber finish composition is present in the aqueous emulsion at a percent actives concentration ranging from about 2 to about 20% actives.

5 15. The process of claim 7 wherein the fiber finish composition further comprises a component selected from the group consisting of a wetting agent, an antioxidant, a biocide, an anti-corrosion agent, a pH control agent, and mixtures thereof.

## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US97/21399

**A. CLASSIFICATION OF SUBJECT MATTER**

IPC(6) :B05D 5/08

US CL :Please See Extra Sheet.

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 106/36, 243; 8/115.56; 252/8.61, 8.63, 8.84, 8.86, 8.91; 427/389.9

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                    | Relevant to claim No. |
|-----------|-------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| Y         | US 5,464,546 A (BIALAS et al) 07 November 1995, col.1, lines 6-10, col. 2, lines 6-49, col. 3, lines 13-17, col. 4, lines 11-21, col. 7, lines 60-67. | 1-15                  |
| Y         | US 5,478,485 A (BIALAS et al) 26 December 1995, Abstract, col. 1, lines 6-12, col. 2, lines 15-42, col. 3 line 1 to col. 8, line 67.                  | 1-15                  |

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |  |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |  |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--|
| <p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"B" earlier document published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> |  | <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"A" document member of the same patent family</p> |  |
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|-----------------------------------------------------------|----------------------------------------------------|
| Date of the actual completion of the international search | Date of mailing of the international search report |
| 12 FEBRUARY 1998                                          | 26 FEB 1998                                        |

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# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US97/21399

## A. CLASSIFICATION OF SUBJECT MATTER:

US CL :

106/36, 243; 8/115.56; 252/8.61, 8.63, 8.84, 8.86, 8.91; 427/389.9