

(19) World Intellectual Property
Organization
International Bureau



(43) International Publication Date
5 February 2004 (05.02.2004)

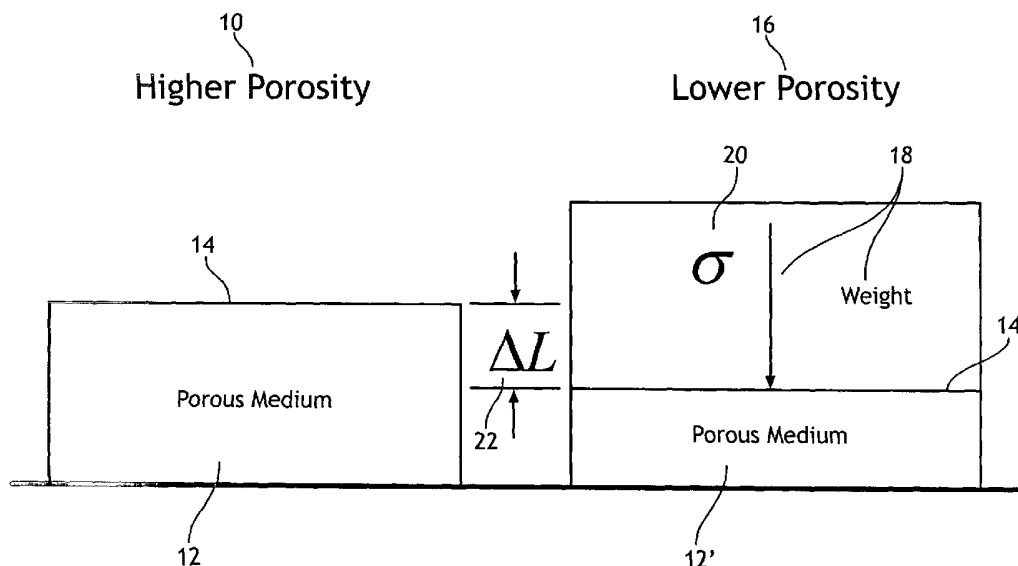
PCT

(10) International Publication Number
WO 2004/011045 A2

- (51) International Patent Classification⁷: **A61L 15/60**
- (21) International Application Number:
PCT/US2003/022423
- (22) International Filing Date: 18 July 2003 (18.07.2003)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
60/399,794 30 July 2002 (30.07.2002) US
10/460,874 13 June 2003 (13.06.2003) US
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- (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VC, VN, YU, ZA, ZM, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).
- Published:
— *without international search report and to be republished upon receipt of that report*

[Continued on next page]

(54) Title: SUPERABSORBENT MATERIALS HAVING HIGH, CONTROLLED GEL-BED FRICTION ANGLES AND COMPOSITES MADE FROM THE SAME



(57) Abstract: The present invention relates to water swellable, water insoluble superabsorbent materials having controlled variable gel-bed friction angles. Controlling the gel-bed friction angle of the superabsorbent materials may allow control of the swelling of the material, the absorbency of the material, and/or the absorbency, resiliency, and porosity of the absorbent composite containing the superabsorbent material. The present invention relates to treatments for superabsorbent materials to manipulate friction angle and new superabsorbent materials having the desired friction angle characteristics. The present invention also relates to absorbent composites employing superabsorbent materials having the desired friction angle characteristics.



For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

SUPERABSORBENT MATERIALS HAVING HIGH, CONTROLLED GEL-BED FRICTION ANGLES AND COMPOSITES MADE FROM THE SAME

BACKGROUND

People rely on absorbent articles in their daily lives.

Absorbent articles, including adult incontinence articles, feminine care articles, and
diapers, are generally manufactured by combining a substantially liquid-permeable
5 topsheet; a substantially liquid-impermeable backsheet attached to the topsheet; and an
absorbent core located between the topsheet and the backsheet. When the article is
worn, the liquid-permeable topsheet is positioned next to the body of the wearer. The
topsheet allows passage of bodily fluids into the absorbent core. The liquid-impermeable
backsheet helps prevent leakage of fluids held in the absorbent core. The absorbent core
10 is designed to have desirable physical properties, *e.g.* a high absorbent capacity and high
absorption rate, so that bodily fluids may be transported from the skin of the wearer into
the disposable absorbent article.

The present invention relates to water swellable, water insoluble superabsorbent
15 materials, which are often employed in an absorbent core (also referred to as an
absorbent composite), in part to help "lock up" fluids entering the core. More specifically,
the present invention pertains to superabsorbent materials having a modified friction angle
measured in a gel-bed of the superabsorbent material. The gel-bed friction angle of the
superabsorbent materials of the present invention is controllable and follows a
20 predetermined pattern. The present invention also relates to use of the controlled gel-bed
friction angle superabsorbent materials in absorbent composites and absorbent articles
incorporating such absorbent composites. Controlling the gel-bed friction angle of the
superabsorbent materials may allow control of phenomena including, but not limited to:
the swelling of the superabsorbent material; stresses experienced by the superabsorbent
25 material and/or other ingredients (*e.g.*, fibers) in an absorbent composite; the permeability
of an absorbent composite containing the superabsorbent material; and/or the
absorbency, resiliency, and porosity of the absorbent composite. The present invention
relates to treatments for superabsorbent materials to manipulate gel-bed friction angle and
new superabsorbent materials having the desired gel-bed friction angle characteristics.

Absorbent composites used in absorbent articles typically consist of an absorbent material, such as a superabsorbent material, mixed with a composite matrix containing natural and/or synthetic fibers. As fluids enter the absorbent composite, the superabsorbent material swells as it absorbs the fluids. The superabsorbent material
5 contacts the surrounding matrix components and possibly other superabsorbent material as it swells. Stresses acting on an absorbent composite comprising the superabsorbent material may act to reduce interstitial pore volume, *i.e.*, space between superabsorbent material, fibers, other ingredients, or some combination thereof (without being bound to a particular analogy, and for purposes of explanation only, think of a force acting on some
10 unit area of a sponge-like material with pores, with the force per unit area - *i.e.*, stress - acting to reduce the thickness of the sponge-like material, and, therefore, the volume of the pores).

As the superabsorbent material swells, it may rearrange into void spaces of the
15 absorbent composite matrix as well as expand readily against the matrix to create additional void space. Also, as the superabsorbent material swells, stresses acting within and/or on the absorbent composite may increase due—at least in part—to expansion of the superabsorbent material, thereby reducing the pore volume between: fibers, superabsorbent material, other ingredients in the absorbent composite, or some
20 combination thereof. The ability to rearrange within the composite matrix, and the magnitude and extent of the stresses acting within and on the composite matrix, depend on several factors specifically including a gel-bed friction angle of the superabsorbent material. In addition, as the superabsorbent material moves within the composite matrix, the superabsorbent material may contact the components, such as fibers and binding
25 materials, of the surrounding matrix. Thus, the frictional properties of the superabsorbent material may influence the ability of the material to swell and rearrange or move within the matrix, as well as the magnitude and extent of the stresses acting within and on the composite matrix.

30 It is often desired that the superabsorbent material be able to rotate and translate within the voids of the absorbent composite to allow the superabsorbent material to swell as close to full swelling capacity as is possible within the matrix. There is a need for a superabsorbent material which may more easily rearrange within the void space of the absorbent composite matrix. There is a need for a way to control the physical mechanics
35 that: allow the superabsorbent material to rearrange within the absorbent composite matrix; reduce or minimize the stresses acting within or on the absorbent composite or its

ingredient(s); and/or reduce the reduction in pore volume that may accompany the build up of said stresses. A novel superabsorbent material fulfilling this need is described in a co-pending application designated as K-C Docket No. 17991A, entitled "Superabsorbent Materials Having Low, Controlled Gel-Bed Friction Angles and Composites Made From The Same," filed on 30 July 2002. This co-pending application is incorporated by reference in its entirety in a manner consistent herewith.

Also, in cases where absorbent composites have initially high porosity or are already fully swollen, it may be desirable to have a superabsorbent material which does not rearrange within the matrix, and thereby maintains porosity and composite permeability by maintaining the free void spaces within the composite matrix.

SUMMARY

We have discovered that superabsorbent materials having controlled gel-bed friction angles meet one or more of these needs. Accordingly, the present invention is directed to superabsorbent materials having controlled gel-bed friction angles. The superabsorbent materials of the present invention have gel-bed friction angles that follow controlled gel-bed friction angle patterns substantially different than gel-bed friction angle patterns followed by conventional superabsorbent materials. The superabsorbent materials of the present invention may be produced using non-conventional manufacturing processes to obtain desired gel-bed friction angles or by treating with additives to increase, decrease, or otherwise control the friction angle of the superabsorbent gel-bed during swelling. Gel-bed friction angle is a property of a gel-bed or superabsorbent material coming from Mohr-Coulomb failure theory.

The superabsorbent material of the present invention may be a water swellable, water insoluble superabsorbent material a first gel-bed friction angle at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material and gel-bed friction angles, at superabsorbent material swelling levels greater than about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material, substantially equal to or greater than the first gel-bed friction angle. The first gel-bed friction angle may be about 30 degrees or greater. The absorbent composite may further comprise a plurality of wettable fibers.

These and other features, aspects, and advantages of the present invention will become better understood with regard to the following description, appended claims, and accompanying drawings.

5

BRIEF DESCRIPTION OF DRAWINGS OF EXAMPLES AND/OR REPRESENTATIVE EMBODIMENTS

10 **FIGURE 1** shows an example of a response of a porous medium to a stress (*i.e.*, a force per unit area) acting on the medium.

FIGURE 2 shows an example of the state of stress of an arbitrary element at equilibrium in a porous medium.

15

FIGURE 3 shows an example of an arbitrary element and the normal forces and shear forces acting on a plane passing through the arbitrary element.

20

FIGURE 4 shows an example of a Mohr Circle on a plot of shear stress (y axis) versus normal stress (x axis).

FIGURE 5 shows an example of a sequence of Mohr Circles corresponding to one possible stress path on a plot of shear stress (y axis) versus normal stress (x axis).

25

FIGURE 6 shows an example of Mohr Circles in relation to a Mohr-Coulomb failure envelope on a plot of shear stress (y axis) versus normal stress (x axis).

FIGURE 7 shows a specific example of Mohr Circles in relation to a Mohr-Coulomb failure envelope on a plot of shear stress (y axis) versus normal stress (x axis).

30

FIGURE 8 shows an example of a friction-angle measuring device, in this case a Jenike-Schulze Ring-Shear Tester available in the U.S. from Jenike & Johanson Inc., a business having offices in Westford, Massachusetts.

35

DEFINITIONS

Within the context of this specification, each term or phrase below will include the following meaning or meanings.

5

“Absorbency Under Load” (AUL) refers to the measure of the liquid retention capacity of a material under mechanical load. It is determined by a test which measures the amount, in grams, of a 0.9% by weight aqueous sodium chloride solution a gram of material may absorb in 1 hour under an applied load or restraining pressure of about 0.3
10 pound per square inch (2,000 Pascals). A procedure for determining AUL is provided in U.S. Patent No. 5,601,542, which is incorporated by reference in its entirety in a manner consistent herewith.

“Absorbent article” includes, without limitation, diapers, training pants, swim wear,
15 absorbent underpants, baby wipes, incontinence products, feminine hygiene products and medical absorbent products (for example, absorbent medical garments, underpads, bandages, drapes, and medical wipes).

“Fiber” and “Fibrous Matrix” includes, but is not limited to natural fibers, synthetic
20 fibers and combinations thereof. Examples of natural fibers include cellulosic fibers (e.g., wood pulp fibers), cotton fibers, wool fibers, silk fibers and the like, as well as combinations thereof. Synthetic fibers can include rayon fibers, glass fibers, polyolefin fibers, polyester fibers, polyamide fibers, polypropylene. As used herein, it is understood that the term “fibrous matrix” includes a plurality of fibers.

25

“Free Swell Capacity” refers to the result of a test which measures the amount in grams of an aqueous 0.9% by weight sodium chloride solution that a gram of material may absorb in 1 hour under negligible applied load.

30

“Gel-bed friction angle” refers to the friction angle of a superabsorbent material in a gel-bed as measured with a Jenike-Shulze ring shear tester or other friction angle measuring technique.

35

“Gradient” refers to a graded change in the magnitude of a physical quantity, such as the quantity of superabsorbent material present in various locations of an absorbent pad, or other pad characteristics such as mass, density, or the like.

"Gel-bed" refers to an amount of superabsorbent material within a container such as a ring shear cell.

5 "Homogeneously mixed" refers to the uniform mixing of two or more substances within a composition, such that the magnitude of a physical quantity of each of the substances remains substantially consistent throughout the composition.

10 "Incontinence products" includes, without limitation, absorbent underwear for children, absorbent garments for children or young adults with special needs such as autistic children or others with bladder/bowel control problems as a result of physical disabilities, as well as absorbent garments for incontinent older adults.

15 "Meltblown fiber" means fibers formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into converging high velocity heated gas (e.g., air) streams which attenuate the filaments of molten thermoplastic material to reduce their diameter, which may be to microfiber diameter. Thereafter, the meltblown fibers are carried by the high velocity gas stream and are deposited on a collecting surface to form a web of randomly dispersed meltblown
20 fibers. Such a process is disclosed for example, in U.S. Patent 3,849,241 to Butin et al. Meltblown fibers are microfibers which may be continuous or discontinuous, are generally smaller than about 0.6 denier, and are generally self bonding when deposited onto a collecting surface. Meltblown fibers used in the present invention are suitably substantially continuous in length.

25

"Mohr circle" refers to a graphical representation of the state of stress within a material subjected to one or more forces. Mohr circles are described in more detail below.

30 "Mohr failure envelope" refers to the failure shear stress at the failure plane as a function of the normal stress on that failure or shear plane. Mohr failure envelopes are described in more detail below.

35 "Polymers" include, but are not limited to, homopolymers, copolymers, such as for example, block, graft, random and alternating copolymers, terpolymers, etc. and blends and modifications thereof. Furthermore, unless otherwise specifically limited, the term

"polymer" shall include all possible geometrical configurations of the material. These configurations include, but are not limited to isotactic, syndiotactic and atactic symmetries.

5 "Superabsorbent" or "superabsorbent material" refers to a water-swella-
ble, water-insoluble organic or inorganic material capable, under the most favorable conditions, of
absorbing at least about 10 times its weight and, more particularly, at least about 20 times
its weight in an aqueous solution containing 0.9 weight percent sodium chloride. The
superabsorbent materials may be natural, synthetic and modified natural polymers and
materials. In addition, the superabsorbent materials may be inorganic materials, such as
10 silica gels, or organic compounds such as cross-linked polymers. The superabsorbent
materials of the present invention may embody various structure configurations including
particles, fibers, flakes, and spheres.

15 "Pattern" or "predetermined pattern," when mentioned in context with gel-bed
friction angle refers to a particular dependence of the gel-bed friction angle on the swelling
level of the superabsorbent material. The pattern of the gel-bed friction angle may refer to
the changes in the gel-bed friction angle of a superabsorbent material as a function of the
swelling level of the superabsorbent material.

20 "Spunbonded fiber" refers to small diameter fibers which are formed by extruding
molten thermoplastic material as filaments from a plurality of fine capillaries of a
spinnerette having a circular or other configuration, with the diameter of the extruded
filaments then being rapidly reduced as by, for example, in U.S. Patent No. 4,340,563 to
Appel et al.; U.S. Patent No. 3,692,618 to Dorschner et al.; U.S. Patent No. 3,802,817 to
25 Matsuki et al.; U.S. Patent Nos. 3,338,992 and 3,341,394 to Kinney; U.S. Patent No.
3,502,763 to Hartmann; U.S. Patent No. 3,502,538 to Petersen; and, U.S. Patent No.
3,542,615 to Dobo et al., each of which is incorporated by reference in its entirety in a
manner consistent herewith. Spunbond fibers are quenched and generally not tacky when
they are deposited onto a collecting surface. Spunbond fibers are generally continuous
30 and often have average deniers larger than about 0.3, more particularly, between about
0.6 and 10.

These terms may be defined with additional language in the remaining portions of
the specification.

35

OVERVIEW OF CONTINUUM MECHANICS, MOHR CIRCLES, AND MOHR-COULOMB FAILURE THEORY

Given that our discovery is described using tools and terminology from mechanics,
5 an overview of continuum mechanics, Mohr circles, and Mohr-Coulomb failure theory is provided for convenience. It should be understood that this overview is for purposes of explanation only—it provides an analytic framework for characterizing the present invention, and should not be viewed as limiting the present invention disclosed herein.

10 Absorbent articles and composites are porous by nature. The open space between the various ingredients that make up the composite (e.g., superabsorbent material and fibers) is commonly referred to as void space or pore space. Pore space acts to store liquids and/or provide a conduit or pathway for transporting liquid throughout the absorbent composite or article. The volume of pore space per unit volume of
15 absorbent composite is commonly referred to as “porosity.” Generally absorbency performance is improved by increasing porosity. For example, permeability of an absorbent composite - *i.e.*, the ability of the composite to facilitate liquid transport - increases with increasing porosity (other factors, such as specific surface area and tortuosity, being equal).

20 The application of a stress to a porous medium, such as an absorbent composite or article, is known to cause a volumetric deformation of the medium as a whole, as well as shear deformation in the case of anisotropic stresses. **Figure 1** depicts an example of a volumetric deformation of a porous medium **12**. The left-most image of **Figure 1** is
25 labeled “Higher Porosity” **10** and shows a porous medium **12** without a weight applied to the uppermost planar surface **14** of the porous medium **12** (with the uppermost planar area having some discrete area). The right-most image of **Figure 1** is labeled “Lower Porosity” **16** and shows the same porous medium **12'** with a weight **18** applied to the uppermost planar surface **14'** of the porous medium **12'**. In response to the placement of
30 the weight **18**, which produces a stress, or normal force per unit area, σ **20**, the thickness decreases (as denoted by ΔL **22**). (Note: for purposes of the present invention, compressive stresses are represented as having positive values.)

For a porous medium **12** made up of individual ingredients such as superabsorbent
35 particles and fibers (e.g., an absorbent composite), the thickness change of the porous medium **12** as a whole, ΔL **22**, likely does not result from a reduction in the individual

dimensions of individual particles and fibers (reductions in these individual thicknesses would likely be small or negligible). Instead, the decrease in the thickness of the porous medium **12** as a whole, ΔL **22**, results from a reduction in porosity (or, analogously, void volume). Accordingly, in the example depicted in **Figure 1**, an increase in stress, or
 5 normal force per unit area, σ **20**, reduces the thickness ΔL **22** of the porous medium **12** as a whole, and reduces the porosity of the porous medium **12**. (Note: If, in **Figure 1**, a fluid in the pores is a compressible gas, then a normal stress acting on the surface of the porous medium **12** would: compress the gas within the pores; or cause a portion of the gas within the pores to exit the porous medium **12**; or some combination thereof. If, in
 10 this same **Figure 1**, a fluid in the pores is an incompressible liquid, then a normal stress acting on the surface of the porous medium **12** would cause a portion of the liquid to exit the porous medium **12**.)

The porous medium **12** of **Figure 1** may be examined further to analyze the
 15 stresses acting on an arbitrary element within the porous medium **12**. **Figure 2** illustrates the state of stress of an arbitrary element **30** - here represented by the face of a cube—at equilibrium (the arbitrary element is within a porous medium **32** being subjected to an external stress σ_{external} **34**). For present purposes, the arbitrary element **30** within the porous medium **32** is treated as a continuum. In **Figure 2**, the state of stress is
 20 represented by two normal components of stress, σ_h **36** acting horizontally on a face of the cube and σ_v **38** acting vertically on another face of the cube, as well as a shear stress τ **40**. The normal components of stress **36** are perpendicular to the faces of the arbitrary element **30**, whereas the shear stresses **40** are parallel to the faces of the arbitrary element **30**.

25 It should be noted that if the shear stresses **40** are zero (*i.e.*, $\tau = 0$), then the two normal stresses **36** are referred to as principal stresses. Furthermore, when $\tau = 0$, then the larger of the two normal stresses **36** is called the major principal stress while the other is called the minor principal stress. For the present discussion, the two stresses are
 30 assumed to be principal stresses, with $\sigma_h \geq \sigma_v$.

There are generally at least two contributions to stress generation that combine to produce principal stresses such as those identified in **Figure 2**. The first is an external stress **34**, possibly non-uniform, acting on the boundary of the porous medium **32**. This
 35 stress is transmitted throughout the porous medium **32** in accordance with well known

force-balance equations. The second contribution arises due to swelling of components that make up the porous medium **32** (e.g., a superabsorbent material). For example, the swelling of blocks, or elements, immediately adjacent to the arbitrary element **30** depicted in **Figure 2**, will cause an “internally” generated stress acting on or along the arbitrary
 5 element **30** as other elements attempt to expand against it and each other.

As stated above, when the stresses acting on an arbitrary element **30**, such as that depicted in **Figure 2**, are principal stresses, there are no shear stresses **40** acting on the faces of the arbitrary element **30**. There is, however, shear stress **40** acting on other
 10 imaginary planes passing through the depicted arbitrary element **30** - planes oriented at some angle α **50** away from horizontal, $0 < \alpha < 90^\circ$, as shown in **Figure 3**. **Figure 3** depicts a major principal stress σ_h **52** acting on a major principal plane **54**, and a minor principal stress σ_v **56** acting on a minor principal plane **58**. A normal stress $\sigma_{n\alpha}$ **60** and a shear stress τ_α **62** act on the imaginary plane **64** oriented at angle α **50** away from
 15 horizontal.

Obtaining the shear and normal forces **62** and **60**, respectively, acting on the arbitrary plane **64** passing through the element **66** depicted in **Figure 3** is simplified by using the graphical approach of the Mohr circle, as illustrated in **Figure 4**. **Figure 4** shows
 20 a plot of shear stress (y-axis) **70** as a function of normal stress (x-axis) **72**. For purposes of the present discussion the principal stresses are assumed to be known (e.g., by calculation or measurement). The x-y coordinates of the minor principal stress σ_v **74** and the major principal stress σ_h **76** lie on the x-axis (i.e., where the shear stress τ **70** is equal to zero). A semi-circle **78** is drawn such that the coordinates of the minor and major principal stresses **74** and **76**, respectively, correspond to the end points of the arc defining the perimeter of the semi-circle **78**. The radius of this semi-circle **78** equals one-half of the difference between the major principal stress σ_h **76** and the minor principal stress σ_v **74**.
 25 By constructing a radial line segment **80** at an angle 2α **82** from the x-axis, with one end of the radial line segment **80** corresponding to the center of the semi-circle **78**, and other end corresponding to a point on the semi-circle arc closest to the major principal stress, both the normal stress, $\sigma_{n\alpha}$ **84**, and the shear stress τ_α **86** are obtained at the intersection
 30 **88** of the radial line segment **80** with the Mohr semi-circle **78**.

Figure 5 depicts one example of stress evolution for a porous medium that employs one or more swelling components (e.g., a particulate superabsorbent material). The y-axis again corresponds to shear stress τ **100**, and the x-axis again corresponds to normal stress σ **102**. If the minor principal stress σ_v **104** acting on an arbitrary element

5 from the porous medium remains unchanged, then stress development (which would accompany, for example, swelling of superabsorbent material) may be viewed as a family of Mohr circles **106**, **108**, **110**, and **112**, all of which have the same minor principal stress σ_v **104**. The progression of Mohr circles **106**, **108**, **110**, and **112**, is commonly referred to

10 as a stress path **114** - more precisely, the line passing through the set of Mohr circles **106**, **108**, **110**, and **112**, at points simultaneously locating the maximum shear stress and mean stress for each Mohr circle **106**, **108**, **110**, and **112**.

The center of each Mohr circle **106**, **108**, **110**, and **112**, which equates to the mean stress, determines the extent of the volumetric deformation of pore space contained within

15 a particular arbitrary element, and may correspond to the approximate stress experienced by superabsorbent materials.

Stresses in a porous medium are not likely to increase indefinitely - rather, failure will take place, accompanied by sliding along particular failure planes (e.g., at the interface

20 between superabsorbent material and fiber; or at the interface between individual particles of superabsorbent material; etc.). The Mohr-Coulomb failure criterion states that a shear force acting on a plane at failure will be linearly proportional to the normal force acting on that same plane, again at failure. Hence, Mohr-Coulomb theory provides a failure limit, or envelope, beyond which stable states of stress do not exist. If a line corresponding to this

25 failure limit is superimposed on a plot of shear stress and normal stress depicting a Mohr circle **106**, **108**, **110**, and **112** (which may be thought of as corresponding to a given state or degree of swelling for a porous medium employing a superabsorbent material), then the Mohr circle **106**, **108**, **110**, and **112**, may only increase in radius (e.g., by additional swelling of the porous medium and/or superabsorbent material employed by the porous

30 medium) to the extent that it becomes tangent to this linear envelope.

Figure 6 depicts a linear failure envelope **120** on a plot of shear stress τ **122** versus normal stress σ **124**. On this plot are depicted two Mohr circles **126** and **128**, with each Mohr circle having a different value of initial stress - that is, two different values of

35 the minor principal stress σ_v **130** and **130'**. The friction angle ϕ **132** and cohesion c **134**

are properties of a particular material (e.g., an absorbent composite comprising fiber and superabsorbent material; a gel bed of swollen, particulate superabsorbent material; etc.). The tangent of the friction angle ϕ 132, which is equivalent to the coefficient of static friction from elementary physics, measures the extent to which an increasing normal force permits a larger maximum shear force. Cohesion c 134 represents the amount of shear stress a material will tolerate before failure in the absence of any normal force on the proposed failure plane. An increase in any one of the three parameters - friction angle ϕ 132, cohesion c 134, or minor principle stress σ_v 130 and 130' - will permit the development of larger stresses in a porous material - i.e., a larger Mohr circle. Friction angle ϕ 132 and cohesion c 134 are properties of the material and may be measured (e.g., using the test and methodology disclosed herein). Figure 6 also depicts the mathematical relationship $\tau_{ff} = c + \sigma_{nff}(\tan \phi)$ 136, which relates friction angle ϕ 132, cohesion c 134, shear stress at failure τ_{ff} 138, and normal stress at failure σ_{nff} 140. (Note: for purposes of this disclosure, σ_{nff} is equivalent to σ_{ff} , with both terms referring to a normal stress acting on the failure plane at failure.) This relationship is described in more detail below in the Detailed Description section.

As stated earlier, it is generally advantageous to minimize or decrease the reduction of porosity, or void volume, that results from the application of a compressive stress to an absorbent article. By choosing materials that limit stress increases (e.g., low, controlled gel-bed friction angle superabsorbent material) the magnitude of porosity reductions may be decreased. For example, low, controlled gel-bed friction angle superabsorbent material will promote the onset of failure before stresses rise to values that cause significant losses of porosity, and therefore permeability. An additional benefit of providing stress relief through low, controlled gel-bed friction angle materials is that such superabsorbent materials will retain a larger portion of their free-swell capacity - since it is well known that superabsorbent capacity decreases with increasing loading. Novel superabsorbent material fulfilling these needs is described in a co-pending application designated as K-C Docket No. 17991A, entitled "Superabsorbent Materials Having Low, Controlled Gel-Bed Friction Angles and Composites Made From The Same," filed on 30 July 2002. This co-pending application is incorporated by reference in its entirety in a manner consistent herewith.

But in some contexts, a superabsorbent having a high gel-bed friction angle is advantageous. For example, if an absorbent composite is in either a highly swollen state

or in a highly porous state, then a superabsorbent having a high gel-bed friction angle may be employed to "lock in" the highly porous structure. The present invention is directed to such novel superabsorbent materials.

5

DETAILED DESCRIPTION OF REPRESENTATIVE EMBODIMENTS

The present invention relates to water swellable, water insoluble superabsorbent materials and the use of the superabsorbents in absorbent composites of absorbent
10 articles.

Absorbent composites of absorbent articles typically contain superabsorbent material, in relatively high quantities in some cases, in various forms such as superabsorbent fibers and/or superabsorbent particles, homogeneously mixed with a
15 matrix material, such as cellulose fluff pulp. The mixture of superabsorbent material and cellulose fluff pulp may be homogeneous throughout the absorbent composite or the superabsorbent material may be strategically located within the absorbent composite, such as forming a gradient within the fiber matrix. For example, more superabsorbent material may be present at one end of the absorbent composite than at an opposite end of
20 the absorbent composite. Alternatively, more superabsorbent material may be present along a top surface of the absorbent composite than along a bottom surface of the absorbent composite or more superabsorbent material may be present along the bottom surface of the absorbent composite than along the top surface of the absorbent composite. One skilled in the art will appreciate the various embodiments available for
25 absorbent composites. The water swellable, water insoluble superabsorbent materials of the present invention may be used in these and other various embodiments of absorbent composites

Absorbent composites typically include a matrix which contains the superabsorbent
30 material. The matrix is often made from a fibrous material or foam material, but one skilled in the art will appreciate the various embodiments of the composite matrix. One such fibrous matrix is made of a cellulose fluff pulp. The cellulose fluff pulp suitably includes wood pulp fluff. The cellulose pulp fluff may be exchanged, in whole or in part, with synthetic, polymeric fibers (e.g., meltblown fibers). Synthetic fibers are not required in
35 the absorbent composites of the present invention, but may be included. One preferred type of wood pulp fluff is identified with the trade designation CR1654, available from

Bowater, Childersburg, Alabama, U.S.A., and is a bleached, highly absorbent wood pulp containing primarily soft wood fibers. The cellulose fluff pulp may be homogeneously mixed with the superabsorbent material. Within the absorbent article, the homogeneously mixed fluff and superabsorbent material may be selectively placed into desired zones of higher concentration to better contain and absorb body exudates. For example, the mass of the homogeneously mixed fluff and superabsorbent materials may be controllably positioned such that more basis weight is present in a front portion of the pad than in a back portion of the pad.

Absorbent composites of the present invention may suitably contain between about 5 to about 95 mass % of superabsorbent material, based on the total weight of the fiber, the superabsorbent material, and/or any other component. Optionally, the mass composition of the superabsorbent material in the absorbent composite may be from about 20 to about 80%. Additionally, the mass composition of the superabsorbent material in the absorbent composite may be from about 40 to about 60%.

Suitable superabsorbent materials useful in the present invention may be selected from natural, synthetic, and modified natural polymers and materials. The superabsorbent materials may be inorganic materials, such as silica gels, or organic compounds, including natural materials such as agar, pectin, guar gum, and the like, as well as synthetic materials, such as synthetic hydrogel polymers. Such hydrogel polymers include, for example, alkali metal salts of polyacrylic acids; polyacrylamides; polyvinyl alcohol; ethylene maleic anhydride copolymers; polyvinyl ethers; hydroxypropylcellulose; polyvinyl morpholinone; polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine; polyamines; and, combinations thereof. Other suitable polymers include hydrolyzed acrylonitrile grafted starch, acrylic acid grafted starch, and isobutylene maleic anhydride copolymers and combinations thereof. The hydrogel polymers are suitably lightly crosslinked to render the material substantially water-insoluble. Crosslinking may, for example, be by irradiation or by covalent, ionic, Van der Waals, or hydrogen bonding. The superabsorbent materials of the present invention may be in any form suitable for use in absorbent structures, including, particles, fibers, flakes, spheres, and the like.

Typically, a superabsorbent polymer is capable of absorbing at least about 10 times its weight in a 0.9 weight percent aqueous sodium chloride solution, and particularly is capable of absorbing more than about 20 times its weight in 0.9 weight percent aqueous

sodium chloride solution. Superabsorbent polymers suitable for treatment or modification in accordance with the present invention are available from various commercial vendors, such as Dow Chemical Company located in Midland, Michigan, U.S.A., and Stockhausen Inc., Greensboro, North Carolina, USA. Other superabsorbent polymers suitable for treatment or modification in accordance with the present invention are described in U.S. Patent No. 5,601,542 issued February 11, 1997, to Melius et al.; U.S. Patent Application Serial No. 09/475,829 filed in December 1999 and assigned to Kimberly-Clark Corporation; and, U.S. Patent Application Serial No. 09/475,830 filed in December 1999 and assigned to Kimberly-Clark Corporation; each of which is hereby incorporated by reference in a manner consistent herewith.

Other examples of commercial superabsorbent materials that may be modified for use in the present invention include polyacrylate materials available from Stockhausen under the tradename FAVOR®. Examples include FAVOR® SXM 77, FAVOR® SXM 880, and FAVOR® SXM 9543. Other polyacrylate superabsorbent materials that may be modified for use in the present invention are available from Dow Chemical, USA under the tradename DRYTECH®, such as DRYTECH® 2035.

The superabsorbent materials of the present invention may be in the form of particles which, in the unswollen state, have maximum cross-sectional diameters typically within the range of from about 50 microns to about 1,000 microns, suitably within the range of from about 100 microns to about 800 microns, as determined by sieve analysis according to American Society for Testing Materials (ASTM) Test Method D-1921. It is understood that the particles of superabsorbent material, falling within the ranges described above, may include solid particles, porous particles, or may be agglomerated particles including many smaller particles agglomerated into particles within the described size ranges.

Absorbent composites may also contain any of a variety of chemical additives or treatments, fillers or other additives, such as clay, zeolites and/or other odor-absorbing material, for example activated carbon carrier particles or active particles such as zeolites and activated carbon. Absorbent composites may also include binding agents, such as crosslinkable binding agents or adhesives, and/or binder fibers, such as bicomponent fibers. Absorbent composites may or may not be wrapped or encompassed by a suitable tissue wrap that maintains the integrity and/or shape of the absorbent composite.

The structure and components of absorbent composites are designed to take up fluids and absorb them. The porosity of the fiber matrix allows fluid to penetrate the absorbent composite and contact the superabsorbent material, which absorbs the fluids. The superabsorbent material swells as the superabsorbent material absorbs fluids. The swelling of the superabsorbent material may be influenced by the external factors such as surrounding matrix material and pressures (*i.e.*, a force per unit area, or stress) from the absorbent article user. The surrounding matrix fibers and/or superabsorbent materials and the pressures on the superabsorbent material may inhibit the swelling of the superabsorbent material, thus stopping absorbency, and thereby the absorbent composite, from reaching full free swell capacity. Also, as described above, stresses acting on an absorbent composite, such as an absorbent composite employing a superabsorbent material, may reduce porosity and/or permeability of the absorbent composite.

The friction angle of the superabsorbent material is an important mechanical property that may affect the ability of the superabsorbent material to move or expand within the absorbent composite matrix, or, alternatively, to reduce such movement where a composite possesses a highly porous, highly permeable structure. As discussed above in the Overview section, friction angle comes from Mohr-Coulomb failure theory, and the tangent of the friction angle is equivalent to the traditional coefficient of static friction. A smaller friction angle may indicate less contact friction between the superabsorbent material and the surrounding matrix, and a greater ability for the superabsorbent material to rearrange within the matrix during swelling so that the superabsorbent material may retain a greater portion of the free swell absorbent capacity. Also, a smaller friction angle may promote failure (*i.e.*, movement between, for example, swollen particles of superabsorbent material; or movement between a swollen particle of superabsorbent material and the surrounding fiber matrix; etc.) at lower levels of stress buildup, thereby reducing losses in porosity and/or permeability in an absorbent composite.

On the other hand, when a superabsorbent material is fully swollen and in a gel-bed or a high porosity absorbent composite, a larger friction angle may indicate more contact friction between the superabsorbent material and the surrounding matrix components which may inhibit the superabsorbent material from rearranging into the voids of the composite matrix, thereby maintaining gel-bed or absorbent composite permeability.

The state of failure between the surfaces of the superabsorbent material and the surrounding components allows the superabsorbent material to rearrange within the wet matrix or a partially swollen gel-bed. As indicated in the Overview Section, Mohr circles may be used to describe the state of stress of a material, such as a wet gel-bed or absorbent composite or porous medium. **FIGURE 7** shows representative Mohr circles **150** and **152** for a typical gel-bed swollen to a particular level. **FIGURE 7** shows Mohr circles **150** and **152** for the superabsorbent FAVOR® 9543 at a 2.0 grams saline solution/gram superabsorbent material swelling level. The larger Mohr circle **152** represents a situation where some pre-consolidation stress is imposed on the gel-bed, and the smaller Mohr circle **150** represents the situation where some major principal stress exists anywhere in the gel-bed while the minor principle stress is zero. Although not shown in **FIGURE 7**, Mohr circles **150** and **152** are produced at each applied normal stress. The state of failure for a superabsorbent material is described by the set of Mohr circles **150** and **152** at failure which together define a Mohr failure envelope. The Mohr failure envelope is often very close to linear, shown in **FIGURE 7** as line **154**, and represents the shear stress at failure, on the failure plane, versus the normal stress acting on the same plane. The linearized failure envelope **154**, often referred to as the Mohr-Coulomb failure criterion, may be represented mathematically by the formula:

$$\tau_{ff} = c + \sigma_{ff}(\tan \phi)$$

where τ_{ff} is shear stress, c is the effective cohesion constant, σ_{ff} is normal stress, and ϕ is the friction angle of the gel-bed or superabsorbent material. The effective cohesion constant is represented on the graph by value **156** and pertains to the cohesion of the absorbent particle to the surrounding medium.

The gel-bed friction angle of the superabsorbent materials of the present invention may be determined using various methods used in fields such as soil mechanics. Useful instruments for determining gel-bed friction angle include triaxial shear measurement instruments, such as a Sigma1, available from GeoTac, Houston, Texas, or ring shear testers such as the Jenike-Shulze Ring Shear Tester, available from Jenike & Johanson, Westford, Massachusetts.

FIGURE 8 shows a partial cut-away schematic of a Jenike-Shulze Ring Shear Tester, designated as reference numeral **170**. The ring shear tester **170** has a ring shear cell **172** connected to a motor (not shown) that may rotate the ring shear cell **172** in direction ω . The ring shear cell **172** and lid **174** contain the superabsorbent material gel-

bed 176 to be tested. The lid 174 is not fixed to the ring shear cell 172 and the crossbeam 178 crosses the lid 174 and connects two guiding rollers 180 and two tie rods 182 to lid 174. For measuring the gel-bed friction angle of swelled superabsorbent material gel-bed 176 the superabsorbent material is swelled outside the ring shear cell 172 and placed in
5 the ring shear cell 172. A predetermined force N may be placed upon the lid 174, and therefore on the superabsorbent material 176, by a weight (not shown). A counterweight system (not shown) may be engaged to test at lower normal pressure. As the ring shear cell 172 rotates in direction ω by the computer controlled motor (not shown), a shear stress is placed on the superabsorbent material gel-bed 176 contacting the ring shear cell
10 172. An instrument connected to the tie rods 182 measures the forces F1 and F2, which are used to determine the shear stress at failure (for a given applied normal stress) of the superabsorbent material gel-bed 176.

In one embodiment of the present invention, superabsorbent material having a
15 high gel-bed friction angle is useful in an absorbent composite which is in a highly swollen state or in a high porosity state. In one embodiment of the present invention, the gel-bed friction angle of the superabsorbent material may be at least about 30 degrees at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent aqueous sodium chloride solution/gram of superabsorbent material (gram/gram) and increases
20 above or remains at about 30 degrees for swelling levels greater than about 5 gram/gram. More suitably, the gel-bed friction angle of the superabsorbent material may be at least about 33 degrees at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent aqueous sodium chloride solution/gram of superabsorbent material and increases above or remains at about 33 degrees for swelling levels greater than about 5
25 gram/gram. More particularly, the gel-bed friction angle of the superabsorbent material may be at least about 38 degrees at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent aqueous sodium chloride solution/gram of superabsorbent material and increases above or remains at about 38 degrees for swelling levels greater than about 5 gram/gram.

When an absorbent composite has high porosity or is in a highly swollen state, the high friction angle of the superabsorbent material may slow and/or inhibit rearranging within the absorbent composite matrix. Slowing and/or inhibiting the rearrangement of the superabsorbent material may maintain an open composite structure, if desired, thereby
35 maintaining a desirable absorbent composite permeability. High gel-bed friction angle superabsorbent materials may be particularly suitable for maintaining highly open

structures when under load. High superabsorbent material gel-bed friction angles may be obtained through manufacturing processes or by treatment of lower friction angle superabsorbent material with various friction angle increasing additives that increase gel-bed friction angle of the superabsorbent material when wet. In one embodiment of the present invention, the cationic polymer friction angle increasing additive chitosan may create a sticky condition between anionic superabsorbent polymers leading to a higher friction angle. Other examples of such friction angle increasing additives include, without limitation, sodium silicate, sodium aluminate, and aluminosilicates.

The amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 1.0% by weight of the swollen or unswollen superabsorbent material or less. Optionally, the amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 10.0% by weight of the swollen or unswollen superabsorbent material or less. Additionally, the amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 100.0% by weight of the swollen or unswollen superabsorbent material or less. The amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 0.001% by weight of the swollen or unswollen superabsorbent material or greater. Optionally, the amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 0.1% by weight of the swollen or unswollen superabsorbent material or greater. Additionally, the amount of gel-bed friction angle increasing additives, surfactants, or emulsifiers may be about 1.0% by weight of the swollen or unswollen superabsorbent material or greater.

Absorbent composites of the present invention may include various controlled gel-bed friction angle superabsorbent materials of the present invention, as well as superabsorbent materials having low gel-bed friction angles, as is described in the co-pending application identified above. The superabsorbent materials with controlled gel-bed friction angles may be homogeneously mixed within the absorbent composite or strategically located within different absorbent composite areas, where the respective controlled gel-bed friction angles are desired.

In one embodiment of the present invention, the gel-bed friction angle of the superabsorbent material may be increased during swelling with a friction angle increasing additive that is located within the superabsorbent material structures in combination with the water swellable, water insoluble polymer. The friction angle increasing additive has a tendency to migrate from within the polymer structure to the surface of the superabsorbent

material as the superabsorbent material swells. In effect, the friction angle increasing additive is not coating, or substantially coating, the superabsorbent material surface when dry and, upon wetting, it migrates to the surface during swelling, thereby causing the gel-bed friction angle of the superabsorbent material to increase. The friction angle increasing additives may be organic and/or inorganic additives, either natural or synthetic.

Small concentrations of emulsifiers and/or surfactants may be used in addition to the friction angle increasing additives, and friction angle increasing additive mixtures, may help increase the gel-bed friction angle of the superabsorbent materials. The emulsifiers and surfactants may increase the miscibility between nonpolar friction angle increasing additives and polar friction angle increasing additives. The emulsifiers and surfactants may also play an integral role in coating the swollen superabsorbent materials. Various emulsifiers and/or surfactants may be used in the present invention depending on the friction angle increasing additive used. Examples of emulsifiers are phosphatidylcholine and lecithin. Examples of liquid surfactants include sorbitan monolaurate, compounds of the TRITON® series (X-100, X-405 & SP-135) available from J.T. Baker, compounds of the BRIJ® series (92 and 97) available from J.T. Baker, polyoxyethylene (80) sorbitan monolaurate, polyoxyethylene sorbitan tetraoleate, and triethanolamine and other alcohol amines, and combinations thereof. When using mixtures of polar and nonpolar compounds, such as friction angle or cohesion value altering additives, emulsifiers, and surfactants, the nonpolar compound may be present in a larger proportion than the polar compound.

In another embodiment of the present invention, the gel-bed friction angle of the superabsorbent material may be increased with a friction angle increasing additive located within the matrix of the absorbent composite. The friction angle increasing additive is in combination with a matrix component, such as coated onto the wettable matrix fibers. The friction angle increasing additive has a tendency to release from the fibers upon wetting and associate with the surface of the superabsorbent material to increase the gel-bed friction angle of the superabsorbent material. Suitably, the friction angle increasing additive debonds with the matrix component at a controlled rate upon wetting, and thereby gradually increases the gel-bed friction angle of the superabsorbent material over a desired time period. The friction angle increasing additives may be organic and/or inorganic additives, natural and/or synthetic materials.

The additives, such as the friction angle increasing additives and friction angle reducing additives, which may alter the friction angle of superabsorbent materials, may be delivered either directly or indirectly to the superabsorbent. Direct delivery could occur through release from the superabsorbent material itself while indirect delivery could occur from fiber or some other component positioned within or adjacent the superabsorbent material and/or the absorbent composite. Furthermore, friction angle altering additives may be delivered gradually over some time period through release from any of the existing components present in the absorbent composite or as the result of some chemical reaction devised to release the friction angle altering additive at the most desirable moment. For example, the friction angle altering additive may be attached to the surface of the superabsorbent material or embedded within its interior, or it may be loaded onto and/or into some other component present in the absorbent composite, including but not limited to the fibrous material. The friction angle altering additive may be available immediately, leading to immediate alteration of the friction angle, or because of a chemical reaction or diffusion or some other mechanism, gradually alter the friction angle in the desired manner at some desired time.

It may be desirable to treat the superabsorbent material, the fiber and/or fibrous matrix, and/or other components that may be used in an absorbent composite with a friction angle altering additive, such as the friction angle reducing additive, the friction angle increasing additive and/or combinations thereof, to provide materials having desired initial friction angles. The material treated with the friction angle altering additive to provide a desired initial friction angle may then be treated with additional friction angle altering additives in accordance with the present invention. The term "substantially" when used herein in regard with friction angle, means within $\pm$ one degree. The term "substantially" when used herein in regard with cohesion value, means within $\pm$ 100 Pascals.

The controlled gel-bed friction angle superabsorbent materials of the present invention may be incorporated into absorbent composites useful in absorbent articles. The various controlled gel-bed friction angle superabsorbent materials of the present invention may be used in various composite structures known in the art, such as described above, including fibrous composites such as meltblown, airlaid, and spunbond composites and foam composites. The superabsorbent materials of the present invention may be formed in various structures in absorbent composites, including particles, flakes, fibers, and spheres.

In accordance with one embodiment of the present invention, a superabsorbent material may comprise a water swellable, water insoluble superabsorbent material. The superabsorbent material may have a first gel-bed friction angle at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material. The superabsorbent material also may have gel-bed friction angles, at superabsorbent material swelling levels greater than about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material, substantially equal to or greater than the first gel-bed friction angle. The first gel-bed friction angle is about 30 degrees or greater.

In accordance with other aspects of the present invention, the first gel-bed friction angle may be about 38 degrees or greater. The water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of natural materials, modified natural materials, synthetic materials, and combinations thereof.

The water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, and combinations thereof. In the alternative, the water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, polyamines, and combinations thereof.

The present invention may further comprise a friction angle increasing additive in combination with the superabsorbent material. The friction angle increasing additive may be selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof. The superabsorbent material may further

comprise a structure selected from the group consisting of particles, fibers, flakes, spheres, and combinations thereof.

In accordance with another embodiment of the present invention, an absorbent composite may comprise a plurality of wettable fibers and a water swellable, water insoluble superabsorbent material in combination with the wettable fibers. The superabsorbent material may have a first gel-bed friction angle at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material. The superabsorbent material also may have gel-bed friction angles, at superabsorbent material swelling levels greater than about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material, substantially equal to or greater than the first gel-bed friction angle. The first gel-bed friction angle may be about 30 degrees or greater.

In accordance with other aspects of the present invention, the first gel-bed friction angle may be about 38 degrees or greater. The water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of natural materials, modified natural materials, synthetic materials, and combinations thereof.

The water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, and combinations thereof. In the alternative, the water swellable, water insoluble superabsorbent material may be selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, polyamines, and combinations thereof.

The present invention may further comprise a friction angle increasing additive in combination with the superabsorbent material. The friction angle increasing additive may

be selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof. The superabsorbent material may further comprise a structure selected from the group consisting of particles, fibers, flakes, spheres, and combinations thereof.

- 5 The absorbent composite may further comprise a friction angle increasing additive in combination with the wettable fibers. The friction angle increasing additive is selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof.

10

Friction Angle Determination

A ring shear testing device such as a Jenike-Schulze Ring Shear Tester apparatus may be used to determine a superabsorbent material gel-bed friction angle. For testing, a
15 sufficient amount (200-1000 grams) of swollen superabsorbent material (e.g., swollen 0-30g/g, or more) is placed within the ring shear cell. For the samples described below, the standard procedure for determining 'yield locus' as described in the manuals 'RST-01.pc, RST-CONTROL' for the Jenike-Schulze Ring shear tester was followed. The specific details for the material preparation and test procedure are given below:

20

The superabsorbent material is swollen to the desired level by 0.9 weight percent aqueous sodium chloride (such as that available from Ricca Chemical Company, Arlington, Texas) in a Kitchen Aid™ blender (model #K5SS, 5 Quart); by first pouring a specific amount of the solution (200 - 1000grams) in the blender bowl (bowl approximate
25 volume: 5 quart) and then adding a predetermined quantity (20 - 600 grams) of dry superabsorbent material while the stirrer is slowly churning the fluid at the lowest speed setting (setting range 1 - 10, where 1 is the lowest and 10 is the highest). This is done so as to distribute the swelling solution uniformly to all the superabsorbent material. When all solution is absorbed by the superabsorbent material (absorption time: 0 - 30 minutes), the
30 bowl is removed from the blender, covered so as to prevent evaporation and allowed to equilibrate for one hour so that the fluid is distributed evenly throughout each particle. The sample is manually mixed every fifteen minutes to ensure that no clumps are formed.

SAP Capacity (g/g)	SAP-Fluid Ratio	Dry Weight Needed (grams)	Saline Weight Needed (grams)	Total Weight SAP-Fluid (grams)	Amount for standard Ring-Cell (grams)
1	1:1	250	250	500	350-450
2	1:2	150	300	450	350-450
5	1:5	80	400	480	400-480
10	1:10	50	500	550	450-550
15	1:15	40	600	640	540-640
20	1:20	30	600	630	550-630

5 If coating is applied to a superabsorbent material, the appropriate coating additive is prepared separately, for example, as described below. The equilibrated (time approximately: 1 hour) and swollen superabsorbent material is coated evenly using a Kitchen Aid™ blender by first introducing the swollen superabsorbent material into the bowl, and then slowly adding the coating additive (addition time: 1 - 30 minutes) while
10 turning the superabsorbent material in the bowl at the lowest speed setting (setting range 1 - 10, where 1 is the lowest and 10 is the highest) with the stirrer at all times. The coated superabsorbent material is allowed to rest for 0 - 30 minutes with manual mixing every five minutes to maintain equal distribution of treatment.

15 The gel-bed friction angle and effective cohesion measurements are determined by using the Jenike-Schulze Ring Shear Tester apparatus. The Jenike-Schulze Ring Shear Tester is used to obtain the gel-bed friction angle values of superabsorbent material gel-beds at various swelling levels. The Ring Shear Tester is operated and calibrated according to the manufacturer's instructions provided. A sample is loaded into the ring
20 shear cell (Volume Ring Cell--standard: 942.48 cm³) while ensuring the superabsorbent gel-bed is distributed evenly (see above table). After one hour of assumed equilibration with 0.9 weight percent sodium chloride solution is achieved, the ring shear cell is filled with the bulk superabsorbent material to be tested (see above table). Even filling may be obtained by removing excess material with a spatula, without compressing the
25 superabsorbent material. The superabsorbent material gel-bed is suitably flush with the top of the ring shear cell. The weight of the filled ring shear cell (without the lid) is

determined on a mass balance and recorded. The samples described below were tested by the ring shear tester control program (RSTCTRL) for 1 - 2 hours. On request from RSTCTRL, the filled shear cell is securely placed on the driving axle. The lid is placed on the ring shear cell and positioned a few degrees counterclockwise from the shear position; the ring shear tester pre-sets this start position. The handle of the counterweight should be on the right side of the crossbeam, and the hook on the crossbeam should be facing the handle. On request from RSTCTRL, the counter weight and the hanger are hooked to the central axis of the crossbeam. The tie rods are attached on each side of the crossbeam, and the ring shear cell is adjusted so that the tie rods are not stressed. The RST-Control offers the possibility to adjust the shear cell with arrow keys: ← → , and using: ↑ ↓ to stop when positioned properly.

During the test procedure, the pressures at which the sample is pre-sheared are read from a control file. In the sample tests described below, the pre-shearing normal pressure is set at 3000 Pascals and the pre-sheared/pre-consolidated gel-bed is then sheared to failure, to obtain the Mohr-Coulomb envelope, at a range of normal pressures ranging from 500 Pascals to 2500 Pascals. Pre-shearing precedes each shearing measurement. Thus, every superabsorbent materialgel-bed is sheared twice at any shearing normal pressure in one experiment. Sometimes the equipment needs to be run in semiautomatic mode and the data point is obtained manually. After the samples below were completed, the results were analyzed using RSV 95, Version 1.0; the software package included with the ring shear tester.

EXAMPLES

To demonstrate aspects of the present invention, superabsorbent material, designated as FAVOR® SXM 9543, available from Stockhausen, Inc., a business having offices in Greensboro, North Carolina, was treated to increase the gel-bed friction angle.

Control

The gel-bed friction angle of the superabsorbent material, untreated FAVOR® SXM 9543, was measured as a control at various swelling levels. The results are summarized in **Table 1**.

Table 1

Swelling level (gram/gram)	2	5	10	15	20
Gel-bed friction angle (degree)	23	15	12	11	12

5

Sample 1

Three amounts of FAVOR® SXM 9543 were swollen to swelling levels of 2 grams, 5 grams, and 10 grams, respectively, of 0.9 weight percent of aqueous sodium chloride solution per gram of superabsorbent material and equilibrated for one hour, as described above. A coating of Sodium Silicate solution, available from J.T. Baker, a business having offices in Phillipsburg, New Jersey, in the ratio of 1.0 gram of additive per 3.0 grams of the swollen superabsorbent material was applied to the swollen superabsorbent material as described above. The gel-bed friction angle was measured as described above. The gel-bed friction angle of the coated superabsorbent material at each of the given swelling levels is listed in **Table 2**.

15

Table 2

Superabsorbent material swelling level	Gel-bed friction angle (in degrees)
2 grams/gram	33
5 grams/gram	31
10 grams/gram	31

20

While the embodiments of the present invention described herein are presently preferred, various modifications and improvements may be made without departing from the spirit and scope of the present invention. The scope of the present invention is indicated by the appended claims, and all changes that fall within the meaning and range of equivalents are intended to be embraced therein.

25

We Claim:

- 5 **1.** A superabsorbent material, comprising:
- a water swellable, water insoluble superabsorbent material; and,
- the superabsorbent material having a first gel-bed friction angle at a
- superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent sodium
- 10 superabsorbent material swelling levels greater than about 5.0 grams of 0.9 weight
- percent sodium chloride solution/gram of the superabsorbent material, substantially equal
- to or greater than the first gel-bed friction angle,
- wherein the first gel-bed friction angle is about 30 degrees or greater.
- 15 **2.** The superabsorbent material of Claim 1, wherein the first gel-bed friction angle is
- about 38 degrees or greater.
- 3.** The superabsorbent material of Claim 1, wherein the water swellable, water
- insoluble superabsorbent material is selected from the group consisting essentially of
- 20 natural materials, modified natural materials, synthetic materials, and combinations
- thereof.
- 4.** The superabsorbent material of Claim 3, wherein the water swellable, water
- insoluble superabsorbent material is selected from the group consisting essentially of
- 25 silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides,
- polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers,
- hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl
- sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted
- starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, and
- 30 combinations thereof.

5. The superabsorbent material of Claim 3, wherein the water swellable, water insoluble superabsorbent material is selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, polyamines, and combinations thereof.

6. The superabsorbent material of Claim 1, further comprising a friction angle increasing additive in combination with the superabsorbent material.

7. The superabsorbent material of Claim 6, wherein the friction angle increasing additive is selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof.

8. The superabsorbent material of Claim 1, further comprising a structure selected from the group consisting of particles, fibers, flakes, spheres, and combinations thereof.

9. An absorbent composite, comprising:

a plurality of wettable fibers; and,

a water swellable, water insoluble superabsorbent material in combination with the wettable fibers and having a first gel-bed friction angle at a superabsorbent material swelling level of about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material and gel-bed friction angles, at superabsorbent material swelling levels greater than about 5.0 grams of 0.9 weight percent sodium chloride solution/gram of the superabsorbent material, substantially equal to or greater than the first gel-bed friction angle,

wherein the first gel-bed friction angle is about 30 degrees or greater.

10. The absorbent composite of Claim 9, wherein the first gel-bed friction angle is about 38 degrees or greater.

11. The absorbent composite of Claim 9, wherein the water swellable, water insoluble superabsorbent material is selected from the group consisting essentially of natural materials, modified natural materials, synthetic materials, and combinations thereof.

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12. The absorbent composite of Claim 11, wherein the water swellable, water insoluble superabsorbent material is selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, and combinations thereof.

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13. The absorbent composite of Claim 11, wherein the water swellable, water insoluble superabsorbent material is selected from the group consisting essentially of silica gels, agar, pectin, guar gum, alkali metal salts of polyacrylic acids, polyacrylamides, polyvinyl alcohols, ethylene maleic anhydride copolymers, polyvinyl ethers, hydroxypropylcelluloses, polyvinyl morpholinones, polymers and copolymers of vinyl sulfonic acid, polyacrylates, polyacrylamides, polyvinyl pyridine, acrylonitrile grafted starch, acrylic acid grafted starch, isobutylene maleic anhydride copolymers, polyamines, and combinations thereof.

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14. The absorbent composite of Claim 9, further comprising a friction angle increasing additive in combination with the superabsorbent material.

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15. The absorbent composite of Claim 14, wherein the friction angle increasing additive is selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof.

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16. The absorbent composite of Claim 9, wherein the superabsorbent material further comprises a structure selected from the group consisting of particles, fibers, flakes, spheres, and combinations thereof.

- 17.** The absorbent composite of Claim 9, further comprising a friction angle increasing additive in combination with the wettable fibers.
- 5 **18.** The absorbent composite of Claim 17, wherein the friction angle increasing additive is selected from the group consisting of chitosan, sodium silicate, sodium aluminate, alumino silicates, and combinations thereof.

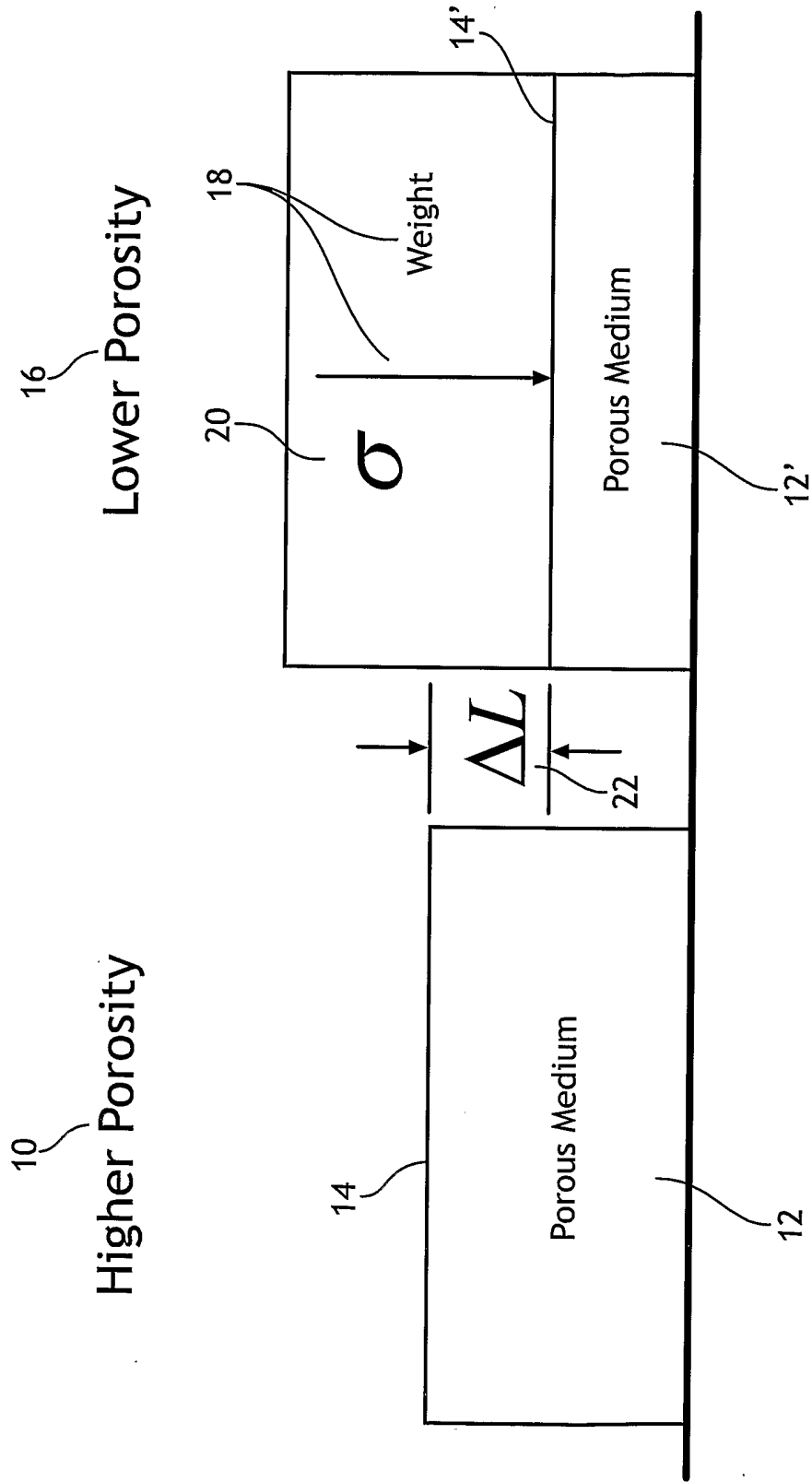


FIG. 1

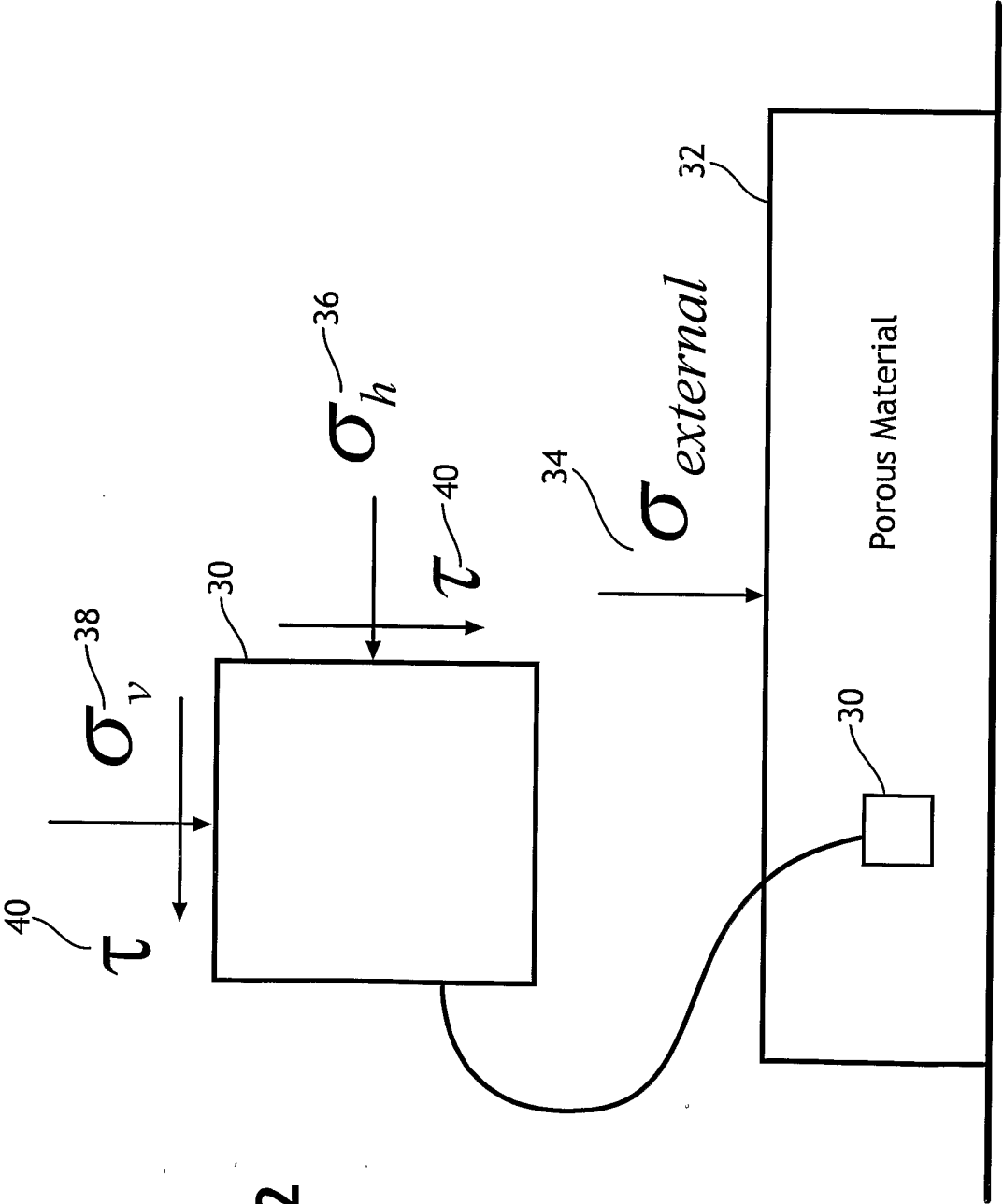


FIG. 2

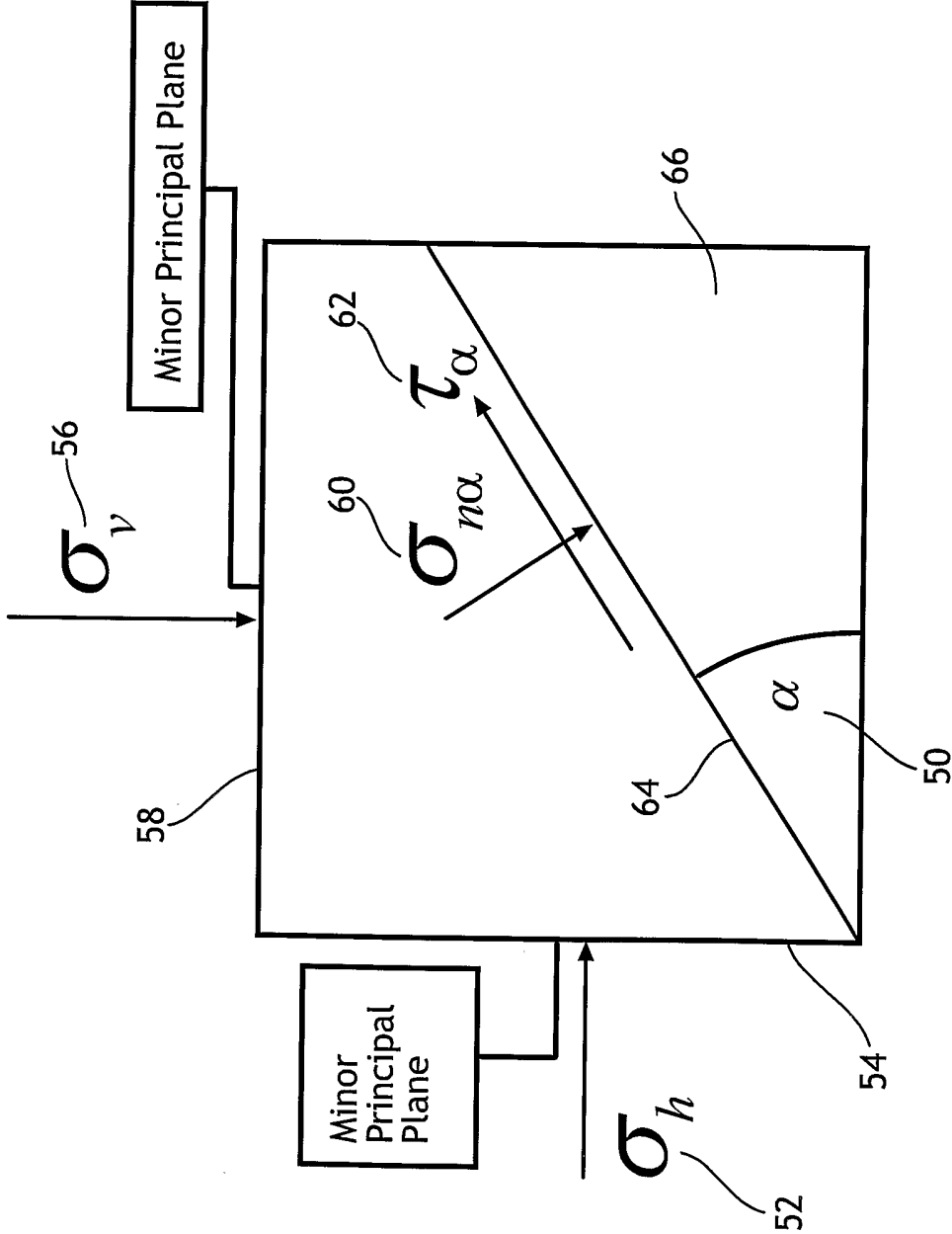
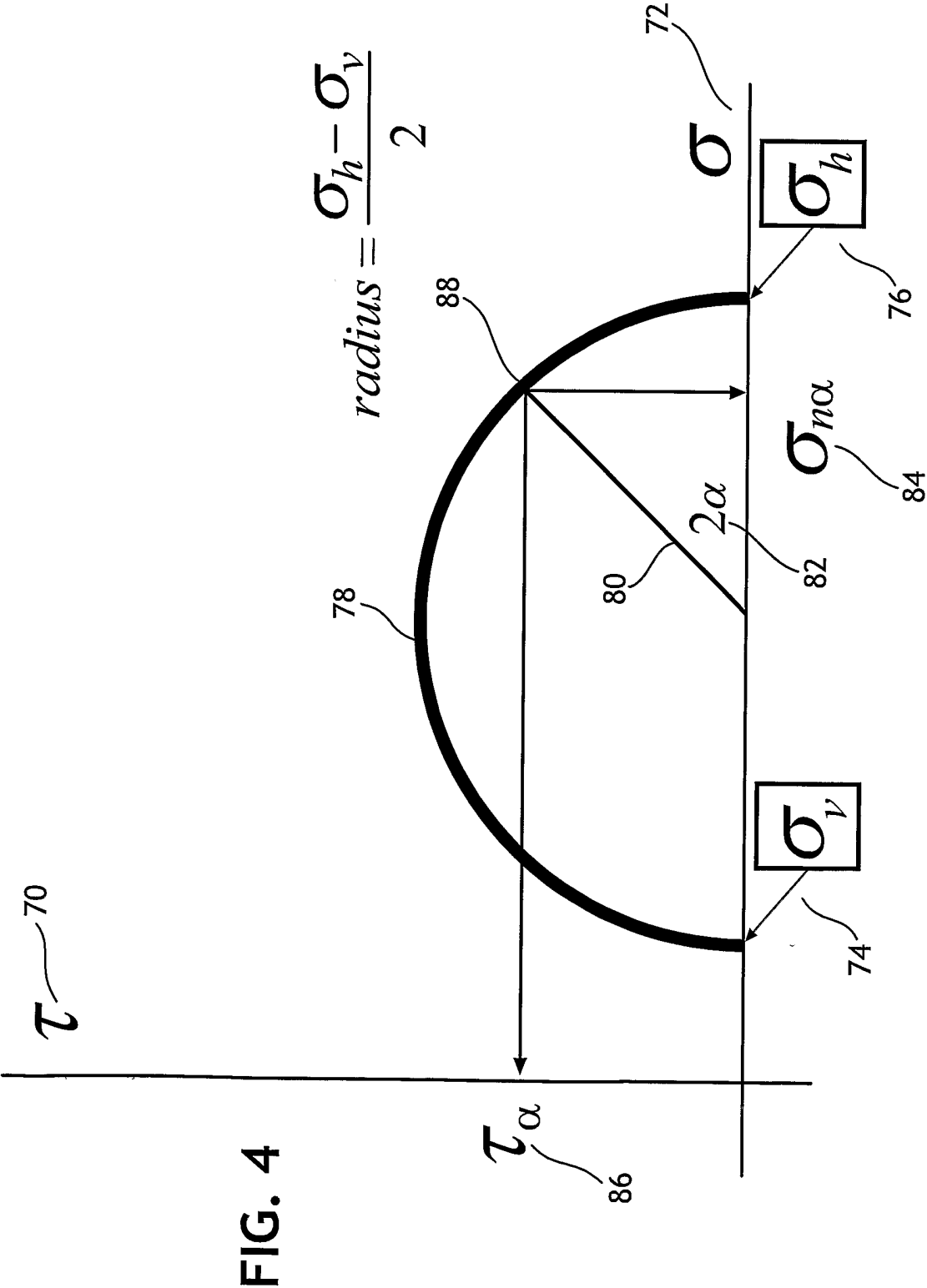


FIG. 3



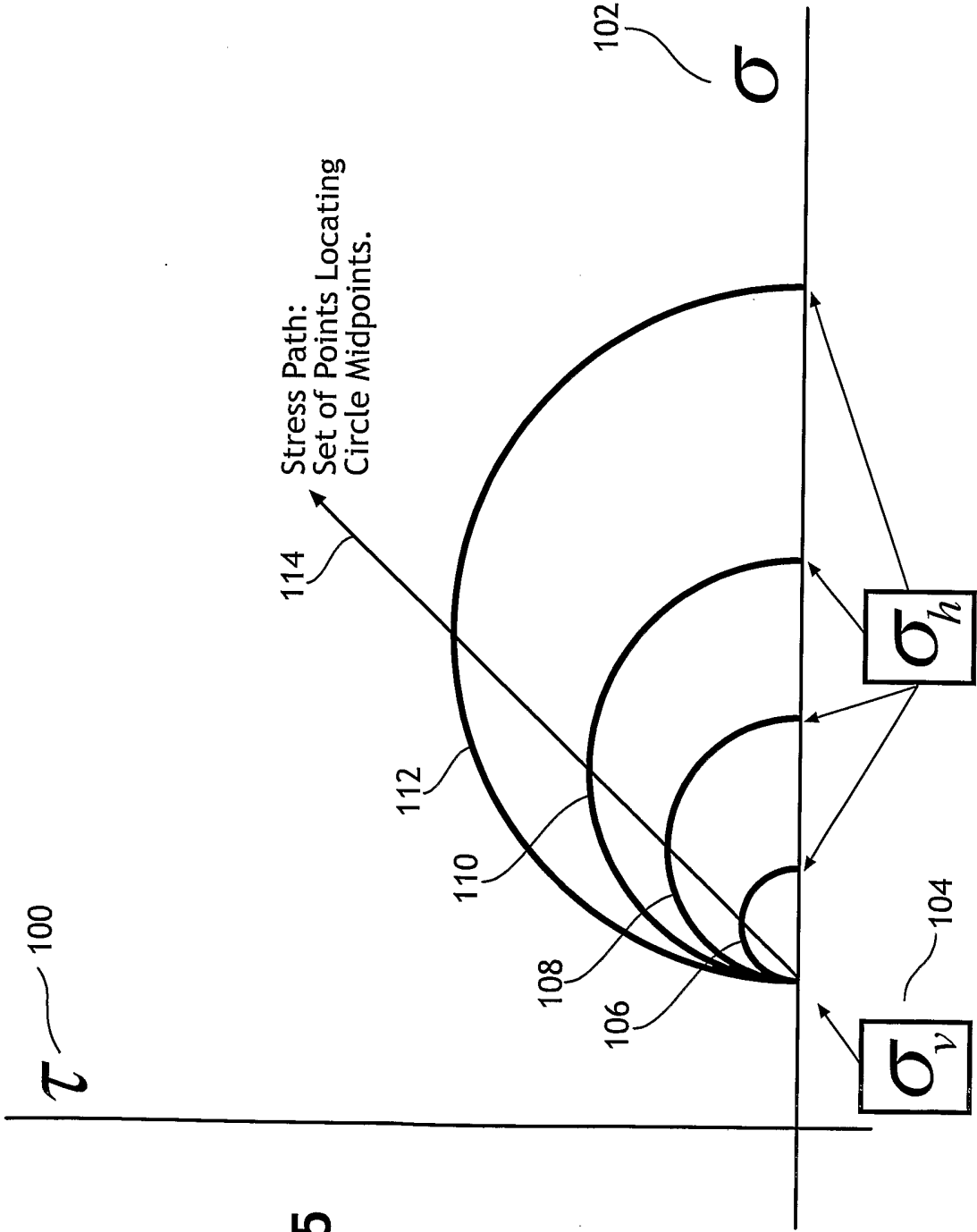


FIG. 5

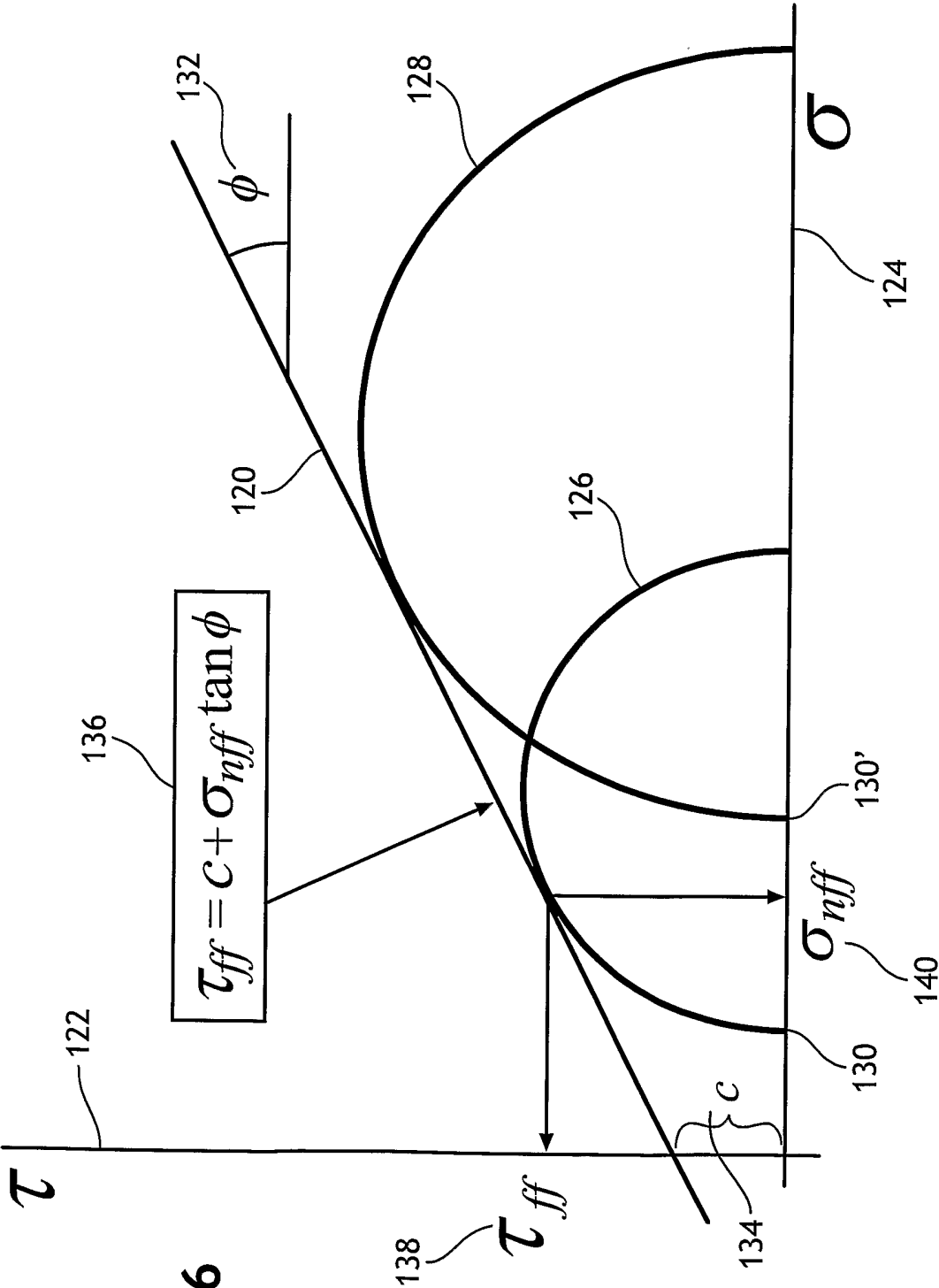


FIG. 6

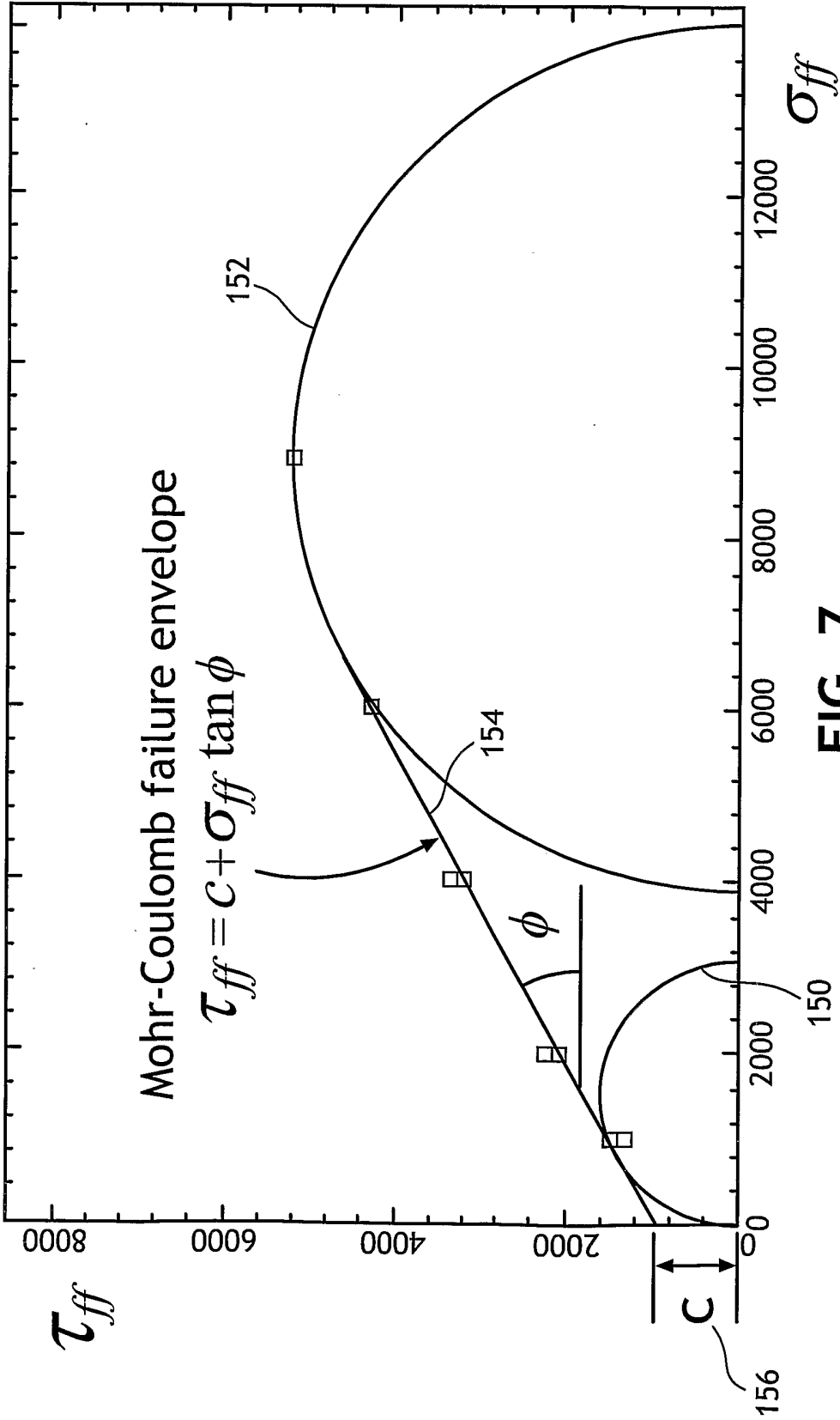


FIG. 7

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