

FORM 1

615650

SPRUSON & FERGUSON

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

APPLICATION FOR A STANDARD PATENT

Minnesota Mining and Manufacturing Company, incorporated in Delaware, of 3M Center, Saint Paul, Minnesota 55144-1000, UNITED STATES OF AMERICA, hereby apply for the grant of a standard patent for an invention entitled:

Encapsulated-Lens Retroreflective Sheeting Having Improved Cover Film

which is described in the accompanying complete specification.

Details of basic application(s):-

Basic Applic. No: Country:

049674

US

Application Date:

13 May 1987

The address for service is:-

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31 Market Street
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DATED this TWENTIETH day of APRIL 1988

Minnesota Mining and Manufacturing Company

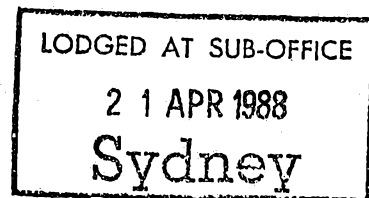
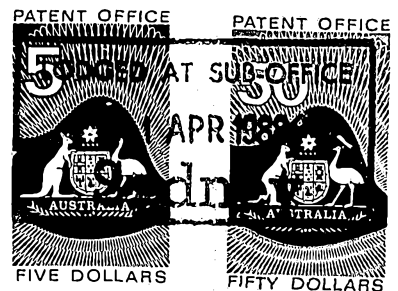
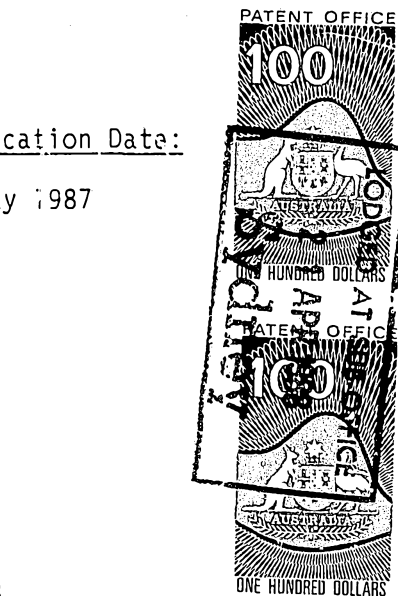
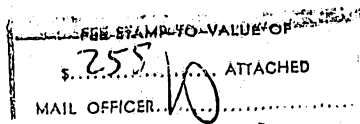
By:

[Signature]

Registered Patent Attorney

TO: THE COMMISSIONER OF PATENTS
OUR REF: 56661
S&F CODE: 58400

5845/2



DECLARATION IN SUPPORT OF A
CONVENTION APPLICATION FOR A PATENTIn support of the Convention Application made for a
patent for an invention entitled:~~42089-AUS-CA~~

Title of Invention

Encapsulated-Lens Retroreflective Sheeting Having
Improved Cover FilmFull name(s) and
address(es) of
Declarant(s)I/We Donald Miller Sell, Chief Patent Counsel,
Minnesota Mining and Manufacturing Company,
of 3M Center, Saint Paul, Minnesota 55144-1000,
United States of America

do solemnly and sincerely declare as follows:-

Full name(s) of
Applicant(s)1. ~~I am/We are the applicant(s) for the patent~~

(or, in the case of an application by a body corporate)

1. I am/We are authorised by MINNESOTA MINING AND MANUFACTURING COMPANY

the applicant(s) for the patent to make this declaration on
its/~~their~~ behalf.2. The basic application(s) as defined by Section 141 of the
Act was/~~were~~ made

Basic Country(ies)

in United States of America

Priority Date(s)

on 13 May 1987

Basic Applicant(s)

by HOWARD R. TOLLIVER, ROGER R. KULT,
TERRY R. BAILEY and ARTHUR D. DICKSONFull name(s) and
address(es) of
Inventor(s)3. ~~I am/We are the actual inventor(s) of the invention referred
to in the basic application(s)~~

(or where a person other than the inventor is the applicant)

3. HOWARD RAYMOND TOLLIVER, ROGER RAYMOND KULT,
TERRY RALPH BAILEY and ARTHUR DONALD DICKSON,

All care of

Minnesota Mining and Manufacturing Company,
3M Center, Saint Paul, Minnesota 55144-1000,
United States of America

(respectively)

~~is/are~~ the actual inventor(s) of the invention and the facts upon
which the applicant(s) ~~is/are~~ entitled to make the application are
as follows:Set out how Applicant(s)
derive title from actual
inventor(s) e.g. The
Applicant(s) is/are the
assignee(s) of the
invention from the
inventor(s)

The said Company is the assignee of the actual inventors.

4. The basic application(s) referred to in paragraph 2 of this
Declaration was/~~were~~ the first application(s) made in a Convention
country in respect of the invention(s) the subject of the application.

Saint Paul

Declared at Minnesota this 11th day of April 1988
U.S.A.

Donald Miller Sell

Signature of Declarant(s)
Donald Miller Sell
Chief Patent Counsel

(12) PATENT ABRIDGMENT (11) Document No. AU-B-15041/88
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- (54) Title
ENCAPSULATED LENS RETROFLECTIVE SHEETING HAVING IMPROVED COVER FILM
- International Patent Classification(s)
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- (71) Applicant(s)
MINNESOTA MINING AND MANUFACTURING COMPANY
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- (56) Prior Art Documents
US 4637950
US 3190178
US 2948191
- (57) Claim

1. An encapsulated-lens retroreflective sheeting characterized in that said sheeting comprises a monolayer of lenses partially embedded in a binder layer, a plurality of specularly reflective layers one each of said reflective layers underlying respective said lenses, and a cover film sealed to said binder layer along a network of interconnecting lines forming hermetically sealed cells within which the lenses are encapsulated and have an air interface, characterized in that:

said cover film comprises polypropylene; or a copolymer comprising a major portion by weight of at least one of ethylene or propylene and a minor portion by weight of at least one comonomer; blends of polyethylene, polypropylene, or copolymers.

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FORM 10

COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952

COMPLETE SPECIFICATION

(ORIGINAL)

FOR OFFICE USE:

Class Int Class

Complete Specification Lodged:

Accepted:

Published:

Priority:

Related Art:

Name and Address
of Applicant:

Minnesota Mining and Manufacturing Company
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Complete Specification for the invention entitled:

Encapsulated-Lens Retroreflective Sheeting Having Improved
Cover Film

The following statement is a full description of this invention, including the
best method of performing it known to me/us

ENCAPSULATED-LENS RETROREFLECTIVE
SHEETING HAVING IMPROVED COVER FILM

Field of Invention

5 The invention concerns encapsulated-lens retro-
reflective sheeting of the type disclosed in U.S. Patent
No. 3,190,178 (McKenzie) having a binder layer in which a
monolayer of lenses such as glass microspheres is partially
10 embedded. A cover film is sealed to the binder layer such
that the lenses are encapsulated within hermetically sealed
cells.

Background Art

15 The earliest retroreflective sheeting had an
exposed-lens construction, and reflex-reflection of light
by such a sheeting was prevented when the lenticular
surfaces of the exposed lenses were covered with water.
This problem was answered by enclosed-lens retroreflective
sheeting in which, as first taught in U.S. Patent No.
2,407,680 (Palmquist et al.), the lenses were embedded
20 within the sheeting which had a flat, transparent cover
film. Such an embedding allowed incident light rays to be
focused onto the specularly reflective layer irrespective
of whether the front of the sheet was wet or dry. The
invention taught in the above-cited McKenzie patent solved
25 the same problem in a different way, namely, by covering
the partially embedded lenses of an exposed-lens type
sheeting with a cover film spaced above the lens. The
binder layer of the sheeting is sealed to the cover film
along a network of interconnecting lines, thus forming a
30 plurality of hermetically sealed cells within which the
lenses are encapsulated and have an air interface. Such
exposed-lens sheeting is sometimes called "encapsulated-
lens retroreflective sheeting".

35 While cover films serve to prevent the direct
wetting of the front surfaces of the microlenses such as by
rain, moisture may still penetrate the cover film, enter

the individual sealed cells, and interfere with effective retroreflection. Therefore, a cover film is preferably substantially impermeable to the migration of moisture therethrough. Furthermore, the cover film should not tend to absorb moisture and become clouded or structurally weakened.

In addition to keeping water from the front surfaces of the lenses and thereby preserving the necessary optical relationships for effective retroreflection, the cover film should protect the underlying elements of the retroreflective sheeting against adverse effects of ultraviolet radiation and moisture penetration, as well as being itself weatherable and tough throughout the expected range of environmental temperatures. It should also have a smooth surface that is highly transmissive to light and should resist dirt accumulation. It is further desired for some uses that the cover film also be receptive to coatings such as marking compositions (e.g., inks).

The overall flexibility and elongation of an encapsulated-lens retroreflective sheeting is substantially affected by the nature of the cover film. Some commonly used cover films, such as polymethyl methacrylate, are not sufficiently flexible and occasionally not sufficiently tough for use in such applications as traffic markers or cones where high flexibility and toughness is preferred.

Summary of Invention

The present invention provides encapsulated-lens retroreflective sheetings having an improved cover film such that such sheetings offer improved flexibility, extensibility, resistance to cracking, and toughness. The cover films provided herein offer excellent impermeability to water, are generally abrasion- and ^{solvent-resistant} ~~solvent-resistant~~, and exhibit good durability to weathering as well as high impact resistance, even at cold temperatures. Furthermore, such cover films are typically less expensive than many conventional cover film materials.



Briefly summarizing, the encapsulated-lens retroreflective sheeting of the invention, like that of the McKenzie patent, comprises a binder layer in which lenses such as glass microspheres are partially embedded and arranged in a monolayer, a specularly reflective layer underlying the microspheres, and a cover film sealed to the binder layer along a network of interconnecting lines forming hermetically sealed cells within which the lenses are encapsulated and have an air interface. The novel retroreflective sheeting differs from that of the McKenzie patent in that the cover film comprises ~~polyethylene, or polypropylene, or a copolymer~~ comprising a major portion by weight of at least one of ethylene or propylene and a minor portion by weight of at least one other comonomer, or blends thereof. Examples of suitable comonomers include acrylic acid, methacrylic acid, vinyl acetate, and ethyl acrylate. Such cover films are sometimes referred to herein as "polyolefin-based" cover films. In some embodiments, the cover film may be multi-layer, e.g., dual layer.

According to the present invention, encapsulated-lens retroreflective sheetings may be made with the cover films disclosed herein and with a variety of binder materials. In some embodiments the cover film or binder material may be primed to achieve desired high adhesion therebetween. In some embodiments, a marking composition (e.g., ink) may be applied to the outside surface of the cover film.

Brief Description of Drawings

The present invention will be further explained with reference to the drawing, wherein:

Figure 1 is a top view of a portion of an encapsulated-lens retroreflective sheeting of the invention;

Figure 2 is a cross-sectional representation of a portion of an encapsulated-lens retroreflective sheeting comprising a cover film of the invention; and

Figure 3 is a cross-sectional representation of a portion of an encapsulated-lens retroreflective sheeting comprising a different embodiment of a cover film of the invention which has a priming layer and a conditioning layer.

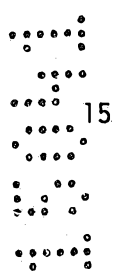
These figures, which are not to scale, are idealized and are intended to be merely illustrative and not limiting.

JMR/998h

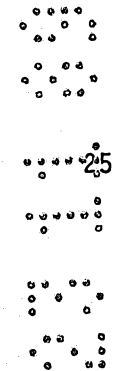


Detailed Description of Invention

As described above, the retroreflective sheetings of the invention comprise a binder layer in which lenses are partially embedded and arranged in a monolayer, a specularly reflective layer underlying the lenses, and a
5 cover film sealed to the binder layer along a network of interconnecting lines forming hermetically sealed cells within which the lenses are encapsulated and have an air interface. Figure 1 is a top view of portion 10 of encapsulated-lens retroreflective sheeting of the invention having cells 12 that each contain several tens or hundreds of individual
10 retroreflective elements. Narrow intersecting bonds or seal legs 14 separate cells 12, hermetically sealing same and bonding the cover film and binder layer together.

The portion 20 of retroreflective sheeting shown in Figure 2 has a monolayer of glass microspheres 22, specularly reflective layers 24, binder layer 26, and dual-layer cover film 28 consisting of outer layer 30 and inner layer 32. On the back side of binder layer 26 is shown disposable low-adhesion carrier web 34 which prevents sticking in the die used to form the network of intersecting bonds 36 between cover film 28 and binder layer 26.

20 To comment on the schematic nature of Figure 2, microspheres 22 will rarely be of essentially uniform size, and the thicknesses of the layers of the sheeting retroreflective sheeting may vary from area to area. Inner layer 32 may be in tangential contact with some of the underlying microspheres or not, depending upon a host of incidental factors.

Portion 120 of the retroreflective sheeting shown in Figure 3 on carrier web 134 comprises microspheres 122, specularly reflective layers 124, binder layer 126, dual layer cover film 128 consisting of outer layer 130, inner layer 132, priming layer 138 and conditioning layer 140.

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The lenses are preferably glass microspheres that have an index of refraction of about 1.9. The diameter of each microsphere is preferably between about 50 and about 200 micrometers. Most preferred are microspheres which have an average diameter of about 65 micrometers. The specularly reflective layer typically comprises a material such as vapor-coated aluminum.

The binder layer typically comprises a flexible and durable polymeric material providing good adhesion to the lenses. The binder layer typically further comprises a whitening agent such as a pigment, e.g., titanium dioxide, to increase the overall whiteness of the sheeting. Alternatively, however, the binder layer may comprise an agent selected to impart a different color thereto.

Best results have been achieved when the cover film has comprised a homopolymer of ethylene or propylene, or a copolymer of monomers comprising a major portion by weight of at least one of ethylene or propylene and a minor portion of at least one other monomer. Substantially transparent blends of polymers with such homopolymers or copolymers may be employed.

Among preferred cover films are ethylene copolymers, a number of which are commercially available at reasonable cost, including:

Supplier	Designation	Comonomer	Weight %	Melt Index
Dow Chemical Company	PRIMACOR 3440	Acrylic acid	9	10
E.I. DuPont	NUCREL	Methacrylic acid	10	35
E.I. DuPont	ELVAX 460	Vinyl acetate	18	2.5
E.I. DuPont	SURLYN 1706	Methacrylic acid	15	0.7
E.I. DuPont	NUCREL 035 ¹	Methacrylic acid	10	35
		Isobutyl acrylate	10	

¹ NUCREL 035 is believed to be a terpolymer. "NUCREL" is a Registered Trade Mark, as is "SURLYN".

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Each of these polymers has excellent flexibility, high impact-resistance, and high toughness at temperatures as low as -40°F (-40°C).

5 Copolymers of monomers comprising by a weight a major portion of at least one of ethylene or propylene and a minor portion of an acid-functional monomer such as acrylic acid or methacrylic acid, which copolymer has been crosslinked by cations such as zinc, sodium, or potassium may be used and are referred to herein as "ionomeric copolymers". Such copolymers typically provide improved
10 tensile strength, abrasion-resistance, and enhanced solvent-resistance. Those ionomeric copolymers that are crosslinked with zinc further are preferred because it is believed to afford the best resistance to weathering. A preferred commercially available ionomeric ethylene
15 copolymer is SURLYN 1706 from E.I. DuPont, a copolymer of ethylene and a minor portion of methacrylic acid ionically crosslinked with zinc ions.

Typically polymers having a melt index of below about 300 are preferred, those having a melt index below about 100 are more preferred, and ^{those} ~~those~~ having a melt index
20 below about 20 are most preferred. Typically materials having a lower melt index tend to be easier to extrude and more resistant to softening at higher temperatures.

An advantage of the materials disclosed herein is that the cover film may be extruded rather than cast from solution, thereby avoiding the cost of solvent and possible pollution from driving off the solvent. Furthermore, extrusion permits faster production rates as compared to the time delay in drying a solvent-cast film.
25

30 Preferably the cover film will be highly transparent, i.e., have a high transmittance of visible light, in order to enhance the retroreflective brightness of the steering. Typically, the cover films of the invention will have a transmittance of at least 75 percent, and preferably at least 85 percent. The copolymers
35 described above typically tend to have high clarity, i.e.,



high transmittance. Cover films comprising a layer of polyethylene will typically have higher clarity if quenched soon after extrusion so as to contain a high portion of amorphous phase polyethylene. Low density polyethylene is typically preferred because it is inherently less crystalline and therefore is more highly transparent than high density polyethylene.

Cover films comprising polyethylene, especially high density polyethylene, may tend to curl and are typically also less preferred for that reason.

Typically, a stabilizer may be incorporated in the cover film. Such a weathering stabilizer may include weathering agents, preservatives, antioxidants, or energy quenchers. For instance, hindered amine light stabilizers have been found useful in the cover films disclosed herein. The cover film will typically also contain an ultraviolet radiation absorber to protect the cover film as well as the underlying elements of the retroreflective sheeting.

Furthermore, a colorant such as a dye may be incorporated in the cover film to impart desired color thereto. Such colorants are preferably selected and/or used in appropriate amounts so as to not interfere with the other desired properties of the cover film, e.g., transmittance or flexibility, or with adhesion of the binder material to the cover film.

Adhesion of the binder material to the inner surface of the cover film is typically enhanced if the binder material is highly compatible with, i.e., has a great affinity to, the inner surface of the cover film at useful sealing temperatures, e.g., about 170°F (75°C). Such compatibility may be achieved if the cover film and binder material are of the same polymer family, i.e., both are olefin-based such as the cover films disclosed herein. Alternatively, the cover film and/or binder layer may be primed, e.g., by corona treatment or with an added layer, to improve the adhesion therebetween, even if the cover film and binder material are of different polymer families.



For instance, aziridine has been added to urethane-based priming layers which are applied as the inner surface of olefin-based cover films such as disclosed herein to provide good adhesion and surface receptivity to a urethane-based binder material. The aziridine-containing priming layer adheres well to the olefin-based cover film and provides a receptive surface to which the urethane-based binder material would not typically adhere well, thereby providing the desired high adhesion between the cover film and binder layer. Aziridine has been similarly used in priming layers used to adhere the cover films disclosed herein to binder materials of a variety of chemical compositions, e.g., in acrylic- and vinyl-based primers.

The cover film materials disclosed herein tend to have greater toughness, flexibility, and extensibility, and to produce encapsulated-lens retroreflective sheetings having such properties, than do materials which have been used in the prior art for transparent cover films.

In some instances it will be desired to provide multi-layer cover films, e.g., dual-layer cover films. In such instances, at least one layer of the cover film will contain one of the aforementioned homopolymers, copolymers, or blends comprising same. For instance, a cover film comprising an inner layer of ethylene/acrylic acid copolymer and an outer layer of polyethylene terephthalate will possess excellent resistance to dirt accumulation and high tensile strength. Alternatively, a multilayer cover film of the present invention may comprise two or more layers that contain one of the aforementioned homopolymers, copolymers, or blends comprising same. For example, a dual layer cover film may comprise an inner layer containing ethylene/acrylic acid copolymer and an outer layer containing Surlyn 1706, an ionomeric copolymer of an ethylene and methacrylic acid crosslinked with zinc, to provide a cover film possessing improved tensile strength and exceptional abrasion resistance.

In such multi-layer cover films, additives such as the stabilizers and colorants may be incorporated in one or more of the individual layers within the cover film. For instance, an ultraviolet radiation absorber may be incorporated in the outer layer of the cover film to protect the inner layer thereof as well as the outer portions of the sheeting.

The materials used in the respective layers of the cover film should be capable of bonding securely together, preferably substantially inseparably so, such that the multi-layer cover film will not delaminate. Multilayer cover films may typically be formed by coextrusion, extrusion of an additional layer onto a previously formed counterpart, or by combination of preformed layers, e.g., heat lamination. Coextrusion is typically preferred because of the strong interlayer bonds which may be attained thereby.

The relative thickness of the member layers within a multilayer cover film of the invention will be determined in part by the needs of the specific application. For instance, a dual-layer cover film may comprise an inner layer containing one of the aforementioned homopolymers, copolymers, or blends and an outer layer containing an exceptionally hard material that resists dirt accumulation, such as unplasticized vinyls. Because such outer layers (i.e., unplasticized vinyls) are typically somewhat inflexible, such a cover film may have a relatively thin outer layer and relatively thick inner layer (for instance, about a 1:9 ratio of thicknesses) to maximize the desired flexibility and toughness provided by the inner layer and yet retain the desired resistance to dirt accumulation and abrasion resistance.

Cover films of the invention will generally be at least about 1 mil (25 microns) thick, and typically be between about 1 and about 10 mils (25 and 250 microns) thick, and more typically between about 2 and about 6 mils (50 and 150 microns) thick.

Sheetings of the invention are typically assembled by preparing a base sheet comprising the binder layer, lenses partially embedded therein, and specularly reflective layer; contacting the ^{preformed}~~performed~~ cover film thereto; and laminating the construction together with heat and pressure to provide the pattern of intersecting bonds between the binder layer and the cover film. Such a method is disclosed in the aforementioned McKenzie patent.

It may be desired to then expose the laminated retroreflective sheeting to an electron beam (i.e., "e-beam") to partially cross-link the cover film to thereby enhance the solvent-resistance and increase the resistance to softening at elevated temperatures of the cover film. Such exposure may also improve the receptivity of the outside surface of the cover film to inks for printing markings thereon, and in the case of multi-layer cover films will typically improve the intra-layer adhesion therein.

It may be desired in some instances to apply a conditioning layer to the outside surface of the cover film, such a layer being receptive to the application of marking compositions, e.g., inks, to enable the application of legends to the face of retroreflective sheeting. Such a conditioning layer is preferably substantially transparent. An example thereof is water borne urethane with an aziridine crosslinker.

The cover films provided herein, particularly the copolymers described above, are typically quite tough and flexible, thus serving to provide great protection and durability to the resultant retroreflective sheeting. Furthermore, the cover films disclosed herein are very impermeable to water and offer high impact resistance even at low temperatures, thereby rendering retroreflective sheetings comprising such cover films to be of great utility in damp environments and for rough use.



The present invention will now be further explained by the following illustrative Examples. In the Examples, all amounts are reported in parts by weight unless otherwise indicated.

5

Testing

The following tests were used to evaluate the encapsulated-lens retroreflective sheetings discussed in the Examples.

10

Tensile and Elongation

ASTM Test Method D882-80a in the down web direction.

Retroreflective Measurements

15

Retroreflective Intensity is measured using a retroluminometer as described in U.S. defensive publication T987,003 at a 0.2° divergence angle and entrance angles of -4° and $+40^\circ$. The Half Brightness Angle (HBA) is the entrance angle at which the retroreflective sample retains one half its original -4° Retroreflective Intensity.

20

Water Absorbtion and Penetration

25

The retroreflective brightness of the retroreflective sheeting in question is measured, and then a sample of the sheeting is adhered to an aluminum panel and immersed in a container of tap water for 24 hours. After removal from the container, the surface of the sample is wiped dry and then the retroreflective brightness measured. Water absorbtion by the cover film and water penetration into the cells of the encapsulated-lens sheeting is indicated by reduction in the retroreflective brightness of the sheeting.

30

Impact Resistance

35

Tested at -10°F (-23°C) according to ASTM D2794-82 using a 0.6 millimeter thickness aluminum panel to

which the retroreflective sheeting is attached. The indentation is in extrusion direction, and the punch has a diameter of 15.9 millimeters.

Water Spray

5 The resistance of the cover film to a high pressure water spray is determined using a HYDRA CLEAN No. 2104ET Model 800-054 Series A with HYDRA No. 7 nozzle, available from Graco, Inc. A sample of the sheeting in question is adhered to an aluminum panel which was then
10 clamped at a 45° angle at a distance of 8 inches (20.3 centimeters) from the nozzle (believed to have a fan angle of 15° and to exert a pressure of 1050 pounds/inch² (7.2×10^6 Newtons/meter²)), and sprayed for 30 seconds. The samples were then visually inspected for damage to the
15 cover film that would indicate failure of the cover film.

Example 1

 A polyethylene-coated paper carrier was heated to about 105°C and then flooded with glass microspheres having
20 an average diameter of about 65 microns and a refractive index of about 1.91. The excess microspheres were removed from the surface of the carrier to leave substantially a monolayer of microspheres thereon, and the carrier and monolayer of microspheres then heated to about 140°C to
25 soften the polyethylene coating such that the microspheres were embedded therein to a depth of about 30 percent of their diameter by gravity and capillary forces. The carrier and monolayer of microspheres were then put into a vacuum chamber and a layer of aluminum deposited thereon to
30 a thickness of about 100 nanometers.

 A binder material comprising 82 parts of a thermoplastic aliphatic urethane binder (Q-THANE P3429, available from K.J. Quinn and Co., Inc. and believed to comprise the reaction product of 1,1-methylene
35 bis(4-isocyanato-cyclohexane), adipic acid, isophthalic acid, and 1,4-butane diol) and 18 parts of rutile titanium

dioxide was dried in a dehumidification drier at 66°C for 16 hours and then extruded onto the polyethylene side of a duplex carrier comprising a 12.5 micron layer of low-density polyethylene and a 12.5 micron layer of polyester. Extrusion conditions were as follows: 171°C, 193°C, 210°C, and 216°C. The extrusion die and plates were set at 204°C and the body of the die at 193°C. Extrusion screw speed and film take-away speed were adjusted to provide a binder film of about 50 microns thickness.

The aluminum side of the monolayer of microspheres was contacted to the binder film and transferred thereto. The transfer conditions included a hot can temperature of 110°C, an applied pressure of 25.2 kilograms/centimeter width, and a line speed of 32 meters/minute. These conditions embed the microspheres to about 20 percent of their diameter in the binder material. The polyethylene-coated paper carrier can then be stripped from the microspheres which remain in the binder film.

Visual examination with a microscope of the carrier revealed that essentially all of the microspheres had been transferred to the binder film, and that essentially all of the aluminum vapor coat that had been deposited on the carrier between individual microspheres remained on the carrier without transfer to the binder film.

A transparent cover film comprising: 97.4 parts of PRIMACOR 3440 (an extrusion grade, thermoplastic, high molecular weight copolymer believed to comprise a major portion of ethylene monomer and a minor portion of acrylic acid monomer, available from DuPont, having a melt flow index of about 10); and 2.6 parts of a stabilizing system (1.0 part of ultraviolet absorber, 1.5 parts of a hindered amine, and 0.1 parts of an antioxidant; the stabilizing system is not believed to affect the strength of the cover film or its adhesion to other materials), was extruded as follows. The stabilized copolymer was extruded from a 6.3 centimeter extruder through a 40.6 centimeter die onto a

biaxially-oriented polyethylene terephthalate (PET) carrier using a single-flighted screw with a compression ratio of 3:1. The extruder temperatures were 190°C, 260°C, 273°C, 264°C, and 264°C for zones 1 through 5, respectively. The extruder neck and die lips were at 270°C. The extruder screw speed was 30 rpm while the film take-away speed was adjusted to provide a film having a thickness of about 100 microns. The extruded film was then wound upon itself into roll form.

One side of the cover film was chosen as the inner surface thereof, corona treated at 0.39 kilowatts per meter² per minute, and then coated with a priming solution comprising 75.0 parts NeoRez R960 (available from Polyvinyl Chemicals Co., believed to comprise water-borne aliphatic urethane), 14.9 parts water, 0.2 parts Witco 3056A bubble breaker, 7.5 parts ethyl alcohol, 0.1 part of a fluorocarbon leveling agent, and 2.3 parts CX-100 (100 percent active polyfunctional aziridine liquid crosslinker from Polyvinyl Chemicals Co.) applied with a 150 line quadrangular knurl coater. The coating was then dried for 10 minutes at room temperature and then cured for 1 minute at 130°C to yield a priming layer having an approximate dry thickness of 2 microns.

The cover film just described was applied to the monolayer of microspheres on the base sheet, primed side down, and sealed thereto along a network of interconnecting bonds under the following conditions:

Embossing can temperature - 360°F (180°C)

Binder preheat temperature - 300°F (150°C)

Embossing can speed - 19 feet/minute (5.8 meters/minute)

Nip roll pressure - 60 pounds/inch-width (69 kilograms/centimeter-width)

After removing the duplex carrier from the binder, an acrylic pressure-sensitive adhesive was applied to the exposed binder layer (side away for the microspheres). Finally the PET carrier was removed from the cover film.

The properties of the resultant retroreflective sheeting are tabulated in Table 1 below.

Example 2

5 Retroreflective sheeting was prepared in Example
1 except the cover film comprised a thermoplastic
homopolymer polyethylene, NPE 3011, believed to be a low
density resin with a specific gravity of about 0.924
gram/cubic centimeter and a melt flow index of about 5.0,
10 available from Enron Chemical Co. The polyethylene was
stabilized with 1.0 part ultraviolet absorber, 1.5 parts of
a hindered amine, and 0.1 parts of an antioxidant. The
cover film was primed with the priming solution described
in Example 1 before being laminated to the base sheet.

15 The cover film just described was applied to the
monolayer of microspheres on the base sheet, primed side
down, and sealed thereto along a network of interconnecting
bonds under the following conditions:

20 Embossing can temperature - 360°F (180°C)
Binder preheat temperature - 300°F (150°C)
Embossing can speed - 9.5 feet/minute (2.9 meters/minute)
Nip roll pressure - 60 pounds/inch-width (69 kilograms/
centimeter-width)

25 The properties of the resultant retroreflective
sheeting are tabulated in Table 1 below.

Example 3

30 Retroreflective sheetings were prepared as in
Example 1 except the cover film was a duplex film
comprising one mil of a polyethylene terephthalate and one
mil of Surlyn 1652. The Surlyn portion of the duplex was
then primed as in Example 1.

35 Sealing conditions were:

Embossing can temperature - 180°C

Binder preheat temperature - 150°C

Speed - 16 feet per minute (4.8 meters per minute)

Nip roll pressure - 69 kilograms per meter width

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The properties of the resultant retroreflective sheeting are tabulated in Table 1 below.

Example 4

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Glass beads or microspheres having an average diameter of about 65 micrometers and a refractive index of 1.91 were flooded onto a polyethylene-paper carrier web which had been heated to about 105°C. Substantially a monolayer of the beads adhered to the polyethylene, and the excess fell off the web. The glass-bead coated polyethylene paper was then heated in an oven at about 140°C to soften the polyethylene so that the glass beads were drawn into the polyethylene to about 30% of their diameter by gravity and capillary forces.

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In a vacuum chamber, aluminum was deposited over the monolayer of glass beads to a thickness of about 100 nm.

25

Onto a 20-micrometer PET support film was extruded a HMW thermoplastic binder film formed from a mixture of pellets of which 69.0 parts were a ethylene/methacrylic acid (EMAA) copolymer and 31.0 parts were a concentrate consisting of 11.9 parts of a polyethylene/vinyl acetate (EVA) copolymer, 18.0 parts of rutile titanium dioxide white pigment, and 1.1 parts of a weathering stabilizer system which in turn consisted of 1 part hindered amine light stabilizer and 0.1 part of antioxidant. The ~~EMMA~~^{EMAA} copolymer comprised 89 parts ethylene and 11 parts methacrylic acid ("Elvax II" 5720 of E.I. DuPont de Nemours & Co.). The EVA copolymer had a melt flow index of 110 and was understood to be a copolymer of 72 parts ethylene and 28 parts vinyl acetate ("Elvax"

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230). The extruder had a diameter of 4.4 cm and a length/diameter ratio of 30:1. The extruder temperature profile (from hopper end to die) was set at 77°, 204°, 149°, 121°C. The polymer transfer tube was set at 132°C, while the film die was set at 143°C. Extruder screw speed was adjusted to 26 rpm while the film takeaway was adjusted to 12.2 m/min. to provide a binder film thickness of about 50 micrometers.

The aluminum side of the monolayer of microspheres was contacted to the binder film and transferred thereto. The transfer conditions included a hot can temperature of 104°C, an applied pressure of 31.6 kg/cm width, and a line speed of 6 m/min. These conditions embedd the microsphere to about 20% of their diameter in the binder material. The polyethylene-coated paper carrier can then be stripped from the microspheres which remain in the binder film.

The cover film of Example 1 was used except that it was not corona treated and primed. Instead the ethylene/acrylic acid cover film was applied to the monolayer of microspheres on the base sheet and sealed thereto along a network of interconnecting bonds under the following conditions:

Surface temperature of embossing can: 174°C,
Embossing speed: 3m/min.,
Nip roll pressure: 21.1 kg/cm width,

After stripping off both PET support films, a pressure-sensitive adhesive layer carried by a protective liner was laminated to the exposed surface of the binder film.

Comparative Example A

A urethane extended polyester with a melamine hardner in solution with solvent is knife coated onto a polyethylene coated paper through 6 mils (150 micrometers)

orifice. This polyester coating is dried in an air forced oven to remove solvents and cure the coating. Oven conditions were 20 minutes at 175°F (80°C) temperature and 10 minutes at 280°F (140°C) temperature. This results in a dried polyester thickness of 2 mils (50 micrometers). This cover film was then sealed to the binder layer of example one using the same sealing conditions. After sealing the duplex carrier web and polyethylene coated paper carrier web are removed and an acrylic pressure-sensitive adhesive and protective liner are applied to the exposed side of the binder layer.

This sheeting is similar to commonly used flexible encapsulated-lens retroreflective sheetings used for substrates which are expected to be impacted by vehicles such as is encountered in construction work zone sites.

Comparative Example B

Encapsulated-lens retroreflective sheeting manufactured using same materials and conditions cited in U.S. Patent 4,025,159, Example 1. This is an example of a commonly used sheeting intended for signing use.

21 4 00 1000

Table 1
Water Absorb./Penet.
(% Brightness
Retention)

Example	Elongation (%)	Tensile (lb/in-width)	Water Absorb./Penet. (% Brightness Retention)	Water Spray (Failure?)	Impact (inch-lbs)	Brightness*
1	135	7.5	100	No	130	215
2	140	6.5	100	No	130	300
3	100	35.0	100	No	130	280
4	350	8.0	100	No	130	260
A	74	7.0	37	Yes	10	250
B	6	35.0	100	No	10	300

*Candella per meter² per lux

Various modifications and alterations of this invention will become apparent to those skilled in the art without departing from the scope and spirit of this invention.

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The claims defining the invention are as follows:

1. An encapsulated-lens retroreflective sheeting characterized in that said sheeting comprises a monolayer of lenses partially embedded in a binder layer, a plurality of specularly reflective layers one each of said reflective layers underlying respective said lenses, and a cover film sealed to said binder layer along a network of interconnecting lines forming hermetically sealed cells within which the lenses are encapsulated and have an air interface, characterized in that:

said cover film comprises polypropylene; or a copolymer comprising a major portion by weight of at least one of ethylene or propylene and a minor portion by weight of at least one comonomer; blends of polyethylene, polypropylene, or copolymers.

2. The sheeting of claim 1 further characterized in that one of said comonomers is acrylic acid, methacrylic acid, vinyl acetate, or ethyl acrylate.

3. The sheeting of claim 1 or 2 further characterized in that said cover film is multi-layer.

4. The sheeting of claim 3 further characterized in that said cover film is dual-layer, comprising:

an inner layer which comprises polypropylene; or a copolymer comprising a major portion by weight of at least one of ethylene or propylene and a minor portion by weight of at least one comonomer; and an outer layer.

5. The sheeting of claim 4 further characterized in that said outer layer comprises a copolymer comprising a major portion by weight of at least one of ethylene or propylene, and a minor portion by weight of at least one comonomer.

6. The sheeting of any one of claims 1-5 further



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characterized in that said cover film is at least about 25 microns thick.

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7. The sheeting of claim 6 further characterized in that said cover film is between about 25 microns and about 250 microns thick.

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8. The sheeting of any one of claims 1-7 further characterized in that the inner surface of said cover film is primed to improve compatibility with said binder layer.

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9. The sheeting of claim 8 further characterized in that said prime comprises a polymeric priming layer.

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10. The sheeting of claim 9 further characterized in that said polymeric priming layer is at least one of the following: of the same polymer family as said binder layer, or comprises aziridine.

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11. The sheeting of any one of claims 1-10 further characterized in that said lenses are glass microspheres.

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12. The sheeting of any one of claims 1-11 further characterized in that said binder layer is white.

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13. The sheeting of any one of claims 1-12 further characterized in that said binder layer contains a whitening agent.

14. The sheeting of any one of claims 1-13 further characterized in that said binder layer is of a different polymer family from said cover film.

15. The sheeting of any one of claims 1-14 further characterized in that said cover film comprises at least one of: a weathering stabilizers or a dye.

16. The sheeting of claim 15 further

characterized in that said weathering stabilizer comprises at least one of the following: antioxidant, energy quencher, hindered amine light stabilizer, or ultraviolet radiation absorber.

17. The sheeting of any one of claims 1-16 further characterized in that said cover film has a transmittance of at least 75 percent.

18. The sheeting of any one of claims 1-17 further characterized in that said cover film has a transmittance of at least 85 percent.

19. The sheeting of any one of claims 1-18 further characterized in that said cover film comprises a conditioning layer on the outside surface thereof.

20. An encapsulated-lens retroreflective sheeting substantially as described herein with reference to and as illustrated by the accompanying drawings.

DATED this TWENTIETH day of APRIL 1988
Minnesota Mining and Manufacturing Company

Patent Attorneys for the Applicant
SPRUSON & FERGUSON

15041/88

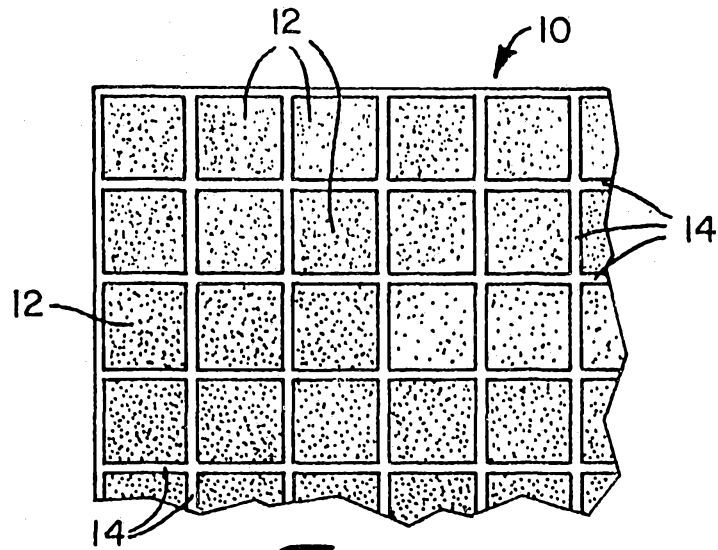


Fig 1

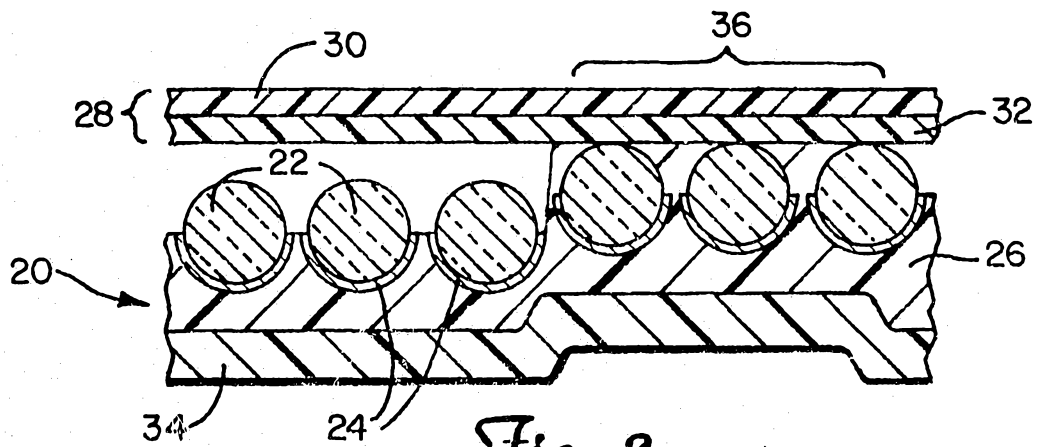


Fig 2

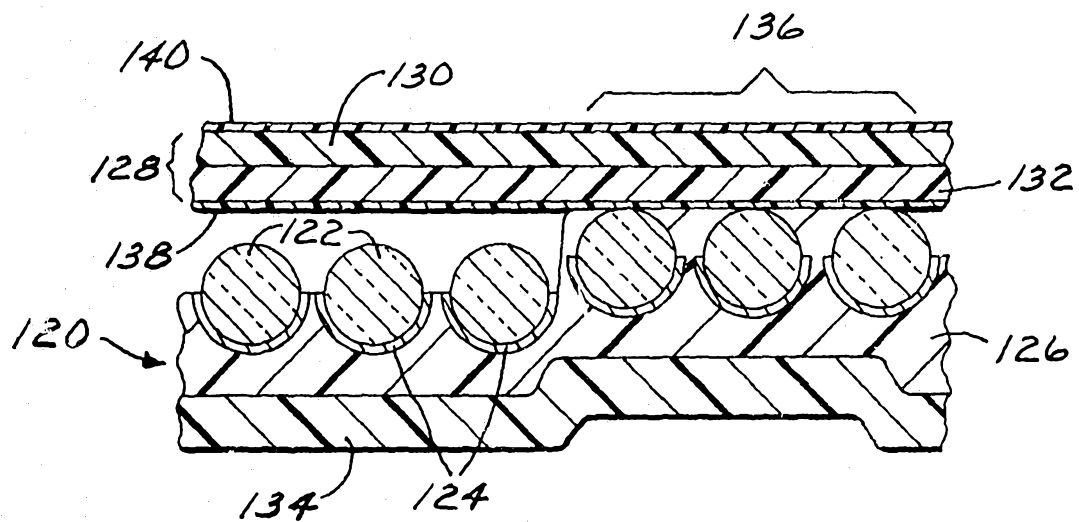


Fig. 3