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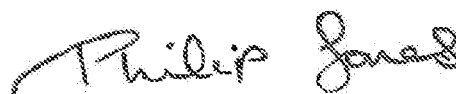
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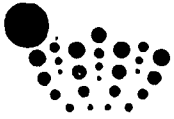
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Patents ADP number (if you know it):

09981424001

3. Title of the invention: OPTICAL COATING

4. Name of your agent (if you have one): J A Kemp & Co

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J A KEMP & CO

Date: 19 August 2010

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Optical Coating

The invention relates to an optical coating, comprising porous silica nanoparticles, or obtained from porous silica nanoparticles, in a suitable binder system, which is transmissive preferably to visible light, and preferably provides antireflective properties, and optionally provides other additional functionality. The coating is particularly, but not exclusively, suitable for application to ophthalmics and eyewear, photovoltaic cells, displays, windows, light emitting diodes and solar concentrators.

Eyewear, solar cells and displays generally consist of an outer substrate exposed to the environment consisting of a sheet of glass or polymer. These typically have a refractive index of 1.5 - 1.7 and reflect about 4 - 5% of incident sunlight on each surface – energy which reduces visibility through the substrate or which is lost to a solar cell. These substrates may be coated with an anti-reflective coating layer that reduces this reflection to less than 2%. Fig. 1 illustrates schematically a conventional single-layer antireflective (AR) coating 1 on a substrate 2. The thickness of the AR coating 1 is h . The reflectance is reduced if the light reflected off the front and back surfaces of the AR coating 1 is arranged to destructively interfere. This is achieved (for normal incidence) if the thickness of the coating 1 is equal to a quarter of the wavelength of the incident light in the medium of the coating, i.e.:

$$h = \frac{1}{4} \frac{\lambda}{n_1}$$

where λ is the wavelength of the light in vacuum, and n_1 is the refractive index of the coating. This assumes that the refractive index n_1 of the coating 1 is less than the refractive index n_m of the substrate 2, such that there is a π phase change of the light reflected at the interface between the coating 1 and the substrate 2. The thickness h may, of course, be any odd integer multiple of one quarter of the wavelength of the light in the coating. For complete destructive interference, the amplitude of the two reflected waves must be equal to each other. This can be achieved if the refractive indices are matched such that:

$$n_1/n_0 = n_m/n_1$$

rearranging this gives:

$$n_1 = \sqrt{n_0 n_m}$$

For air $n_0 = 1$, and for glass $n_m = 1.5$, which gives the ideal refractive index of the coating as $n_1 = 1.22$.

In eyewear applications AR coatings are used to increase transmission of light and reduce reflections within the inner lens surface that can be damaging to the eyes of the wearer.

- 5 In display applications, AR coatings are used to reduce reflectance that diminishes the viewability of the display, i.e. to reduce glare. Another desirable property of such coatings is a reduction in reflectance over a wide viewing angle. In such cases, the AR coating is primarily applied to plastic substrates although glass may also be used.

10 However, there are a number of problems with conventional AR coatings. There is difficulty in finding suitable coating materials with the desired low refractive index. The coatings are typically applied by techniques such as chemical vapour deposition (CVD) or physical vapour deposition (PVD) which require costly processing and are difficult to use with substrates other than glass, such as plastic windows for solar concentrators. However the relatively inert surface chemistry of typical polymeric materials used for these components
15 can lead to poor adhesion of subsequently coated layers.

The above analysis shows that optimal antireflectance properties are only achieved at one wavelength for one particular angle of incidence; at other wavelengths and angles of incidence, the antireflectance deteriorates and so the efficiency of the solar cell or the readability of the display is reduced. Broadband AR coatings can be achieved by using
20 multiple layers of differing refractive index, but this increases the complexity and cost of manufacture, which makes the solar cells or displays more expensive and less economically viable. There can also be problems with applying AR coatings in addition to other functional coatings that may desirably be present on the solar cell, such as so-called 'self-cleaning' coatings.

- 25 It is proposed that an effective broadband single layer antireflective coating can be formed by a simple low temperature wet chemical coating technique such as spin, dip, web or roll coating and such an anti-reflective coating would consist of porous silica nanoparticles of low refractive index and a binder material used to provide mechanical strength.

30 For instance, US 2009-0220774A1 proposes using mesoporous silica nanoparticles consisting of a regular hexagonal array of pores formed by the use of a cationic surfactant which is used to template the pore structure. These particles are applied to a substrate before the coating is

baked, preferably at a temperature of higher than 500°C to remove the surfactants and densify the layer. However, this does not allow use on polymer substrates due to the high baking temperature. The lack of a binder system and the degree of sintering of the nanoparticles due to the baking reduce the mechanical flexibility of the system and its ability to withstand flex and impact.

JP 2009-40967 also proposes using a mesoporous silica nanoparticle system in which the particles are formed with a regular array of pores templated by a quaternary ammonium salt cationic surfactant. After particle formation the surfactant is removed by washing in acid solution and an anti-reflective coating is formed by dispersing the particles in a binder system and depositing them on a suitable substrate prior to drying and curing the binder system. The regular structure of pores in the nanoparticle and the nature of the surfactant make complete removal of the surfactant easier but this leaves open to the ingress of the binder and solvent into the pore system by capillary forces which degrades the anti-reflective performance by increasing the refractive index of the particles formed.

It is an object of the present invention to alleviate, at least partially, some or any of the above problems.

Accordingly, the present invention provides an optical coating comprising a binder and a plurality of porous silica nanoparticles in which the pores are randomly oriented, and wherein said pores have an internal surface at least partially comprising a hydrophobic polymer layer

Another aspect of the invention provides a method of fabricating an optical coating, said method comprising:

providing a blend of porous silica nanoparticles in which the pores are randomly oriented, and wherein said pores have an internal surface at least partially comprising a hydrophobic polymer layer, in conjunction with a binder and a solvent;

coating the particles, binder and solvent as a layer onto a substrate; and

removing solvent from the layer to form the optical coating.

According to a preferred embodiment of the present invention, the optical coating comprises a binder-nanoparticle composite film in which porous silica nanoparticles have randomly oriented pores, a tortuous pore path being preferred for reducing liquid ingress, which pores are coated with a thin (e.g. monolayer) of polymer – typically polystyrene. Substantially all

of the pores have a mean size in the range of 1-10 nm, more preferably the mean size of each pore is in the range of from 1 to 3 nm. The pores are primarily air filled except for this thin polymer internal surface. There is also a layer of polymer on the outer surface which acts to link the particles together when deposited in a thin film prior to the majority of inter-particle space being filled with the desired binder. Due to the hydrophobic nature of the internal pore structure and its random nature there is substantially no binder ingress into the pores and the refractive index of the particles is maintained at less than 1.20.

A binder material, which may be either inorganic or organic, between the particles also acts to provide mechanical strength to the film and the similarity of properties between the binder and the underlying substrate eliminate brittle and interface film failure even under loads that induce significant distortion to the coating. This may include use of a suitable primer on the substrate or hardcoat to enhance interface adhesion.

Preferably the film can exhibit hardness of typically greater than 0.7 GPa, or more preferably greater than 1.0 GPa. Also preferably, the coating has an elastic modulus greater than half and less than twice the elastic modulus of the underlying substrate. In this way the elastic modulus can match the underlying substrate, which indicates that the film is capable of significant flex. The preferred coating embodying the invention will flex without brittle failure of the film to greater than ten times the film thickness on flexible substrates, for example polymer substrates. This flex is even seen when a film comprising an inorganic binder is used on a polymer substrate. This surprising result is another aspect to the composite porous silica-organic structure of the nanoparticles.

The binder material will typically have a refractive index of greater than 1.5. The refractive index of the mixture of the particles and the binder system can therefore be tailored to a specific substrate. This allows the system to optimise the refractive index of the coating to minimise the reflectivity of the optical coating in the case of an anti-reflective coating film.

In the present specification, the term "optical" is used, for example in "optical coating"; however, this term is not intended to imply any limitation to visible light only. The invention may, if required, be applied to other parts of the electromagnetic spectrum, for example including at least ultraviolet (UV) and infrared (IR). The coating of the invention is also referred to as a film in some contexts.

Embodiments of the invention will now be described, by way of example only, with reference to the accompanying drawings, in which:

Fig. 1 is a schematic illustration of a conventional uniform-thickness, single-layer AR coating provided on a substrate;

5 Fig. 2 is a Scanning Electron Micrograph of a cross-section of the optical coating on a glass substrate for solar cell applications;

Fig. 3 is a reflectance curve in the visible wavelength range showing the anti-reflective performance of the optical coating of Fig. 2;

10 Fig. 4 is a Scanning Electron Micrograph of a cross-section of the optical coating on a silicone hardcoated polycarbonate substrate for use in ophthalmic applications; and

Fig. 5 is a transmission curve showing the anti-reflective performance of the optical coating of Fig. 4.

One embodiment of the optical coating relates to the use of porous nanoparticles in an antireflectance coating in conjunction with a binder system. The particles have an open or porous structure. Porous particles are used as antireflectance coatings because the porous nature of the material naturally reduces the refractive index (i.e. the refractive index becomes an average of that of air and the material of the particles). As such they may be applied to a surface and fulfil the requirements of having a refractive index close to halfway between glass and air.

The particles are mesoporous (with pore diameters greater than 2 nm, but typically not greater than 10 nm) or microporous (with pore diameters less than 2 nm), or a mixture of the two. In general, the particles are less than 100 nm in maximum dimension and have a random pore structure with mean pore diameter less than 10 nm.

25 The porous particles comprise at least one of mesoporous particles and/or microporous particles in which the pores are arranged randomly and the pores are internally coated with a hydrophobic polymer. The polymer can be in a monolayer, and can comprise, for example polystyrene or poly vinyl butadiene.

In one preferred embodiment, the porous particles comprise mesoporous silica particles with a size less than 50nm and a pore diameter of 1-3nm and an internal organic hydrophobic polymer coating of less than 30 %.wt of the particle.

5 Most preferably, the porous particles are in the size range 20-30nm in order to reduce any surface roughness of the film to less than 30nm.

SYNTHESIS OF PARTICLES FOR USE IN EMBODIMENTS OF THE INVENTION

10 Porous silica nanoparticles are typically prepared by the hydrolysis of an alkoxy silane (such as tetramethylorthosilicate and tetraethylorthosilicate) followed by co-condensation of the hydrolysed precursor to produce an inorganic silica polymer. To produce particulate structures the reaction is catalysed by the presence of a base, which accelerates the condensation reaction. Any suitable base may be employed, for instance ammonia, NaOH or KOH. Thus the reaction is typically performed in an alkaline solution, which is typically an aqueous solution of the base. Typically this reaction would result in large, dense spherical silica particles.

15 The inclusion of a polymeric templating agent results in structural modification of the particle and the development of a randomly oriented pore structure. For example, if polystyrene is polymerised in the same solution as the above reaction, then the space occupied by the organic polymer cannot be occupied by the silica, and hence the silica grows around the polymer, resulting in an intimately mixed organic/inorganic particle. Removal of the templating polymer, by a solvent that dissolves polymer and not silica, results in a silica particle with pores resulting from the polymer removal. Polymer removal is never complete because the surface energy increase of completely removing the polymer from the silica surface is too large. Hence a degree of polymer coating is retained within the silica pores on the internal surfaces of the pores.

25 The overall particle size is controlled by forming an oil in water emulsion. The emulsion droplets act to halt growth of the particle beyond the domains of the droplet. The droplet size is controlled by the ratio of oil, water and emulsifying agent type and concentration. Under appropriate conditions, particle size and size distribution can therefore be kept to a size of 20-30 nm.

The porous silica particles, fabricated as described above, are such that the pore structure is randomly oriented and the internal surface of the pores is coated with a hydrophobic layer and the external surface of the particle is hydrophilic.

OPTICAL COATING

5 The particles are used to create a layer on a substrate, such as glass or polymer, which layer has a mean thickness in the range from 75 to 500 nm, with a surface roughness in the range from 2 to 50 nm and a refractive index in the range of 1.1 to 1.4. A more preferred value for the thickness is in the range from 100 to 200 nm. A more preferred surface roughness is in the range from 5 to 30 nm, most preferably 10 to 30 nm.

10 The optical coating is obtained by formulating the particles above in a binder system and a solvent.

Examples of the binder system comprise at least one of silicate, silica, silicone based polymer, siloxane based polymer, acrylate based polymer, cellulose, cellulose derivatives, or vinyl alcohol, dispersed within a solvent.

15 The solvent system in this preferred embodiments is primarily alcohol based, e.g. based on at least one of ethanol, propanol or butanol, but may also contain other components such as water or acid or silicone, which control the viscosity of the solution and the dispersion of the particles.

20 The formulation described above is deposited by standard wet chemical coating techniques, including but not limited to spin coating, dip coating, roll to roll coating and webcoating on a substrate.

The substrate is, for example, one of glass, quartz, polycarbonate, silicone hardcoated polycarbonate, acrylate coated polycarbonate, polymethyl methacrylate, polyethylene terephthalate (PET), polyethylene naphthalate (PEN), or cellulose triacetate (TAC).

25 The formulation is dried and cured on the substrate to form the optical coating. The drying is a process to remove the solvent. The drying can be performed simultaneously with the curing or can constitute a separate process. The curing is performed by maintaining the temperature in the range of from 50 to 250°C, more preferably from 80 to 140°C; alternatively UV curing is performed at ambient or elevated temperature.

The combination of the optical coating and the underlying substrate are matched by manipulation of the ratio of particles to binder and by the choice of binder. This matching allows the coating to flex under continuous pressure or during an impact, for example from a sand particle hitting the surface whilst maintaining the hardness of the optical coating.

5

EXAMPLES

Example 1 – Optical coating on glass substrate for solar cell applications

A solution of 1.4% w/v mesoporous silica nanoparticles (as described above) in methanol is used as a source of particles (Solution A). The size range of the mesoporous silica particles is 10 20 - 30 nm. A binder solution comprising 100µl tetraethyl orthosilicate (TEOS), 2ml isopropanol (IPA) and 50µl hydrochloric acid is prepared (Solution B). Glass substrates are prepared by washing in acetone at 60°C for 10 minutes, IPA at 60C for 10 minutes and are then dried. The dimensions of the substrates are 25 mm x 25 mm.

The anti-reflection coating is prepared using a spin coater. A substrate is spun at 4200rpm 15 and 270µl of Solution B is deposited on the substrate which continues spinning for 25 seconds. Following this 270µl of Solution A is deposited on the substrate which is spun at 4200rpm for 25 seconds. These two deposition steps are then repeated to give a final coating with the correct optical and mechanical properties.

The structure is shown in cross-section in Figure 2, in which the optical coating (1) is on the 20 glass substrate (2); the reflectance properties in comparison to an uncoated glass substrate are given in Figure 3. As can be seen, the reflectance for all wavelengths of visible light in the range from 390 to 750 nm is less than 2%, and in fact less than 1.5%. These low reflectances can also be achieved for wavelengths in the range of from 300 to 1900 nm.

Example 2 – Silica particles and silicone binder on polycarbonate substrate for ophthalmic 25 applications

1g of a solution (4.7% wt in ethanol) of mesoporous silica nanoparticles (as described above), is diluted with 2.35ml of Isopropanol to obtain a 1.4% wt solution (Solution A). A thermally curable hardcoat MP1154 from SDC Coatings (Anaheim, CA) is used as a binder. A binder solution of 1ml MP1154 is diluted with 1ml of Isopropanol to obtain a 10 wt% binder

solution (Solution B). 3.4ml of Solution A is mixed with 0.6ml Solution B to obtain the anti-reflective coating solution (Solution C). Prior to coating a silicone hardcoated polycarbonate lens is primed using PR-1165 from SDC Coatings (Anaheim, CA). 500µl of Solution C is deposited on a silicone hardcoated polycarbonate lens on a spin-coater which is then spun at 4000rpm for 1 minute. The resulting optical coating is then cured in air at 129 °C for 4 hours to produce the coating structure shown in Figure 4, comprising the optical coating (1), the silicone hardcoat (2) and the polycarbonate substrate (3). The transmission of the substrate with and without the anti-reflection layer (optical coating) is shown in Figure 5; the greater transmission with the coating demonstrates the reduction in reflection.

10 Example 3 – Measurement of hardness and elastic modulus of coatings on glass and polycarbonate substrates.

Coatings of thickness 150nm comprising of 20-30nm mesoporous silica particles and a binder of silicate are formed on quartz and a silicone hardcoated polycarbonate substrate in accordance the procedure given in Example 1. The coatings are analysed using a

15 Nanoindenter (Micro Materials UK) in order to ascertain the hardness and modulus of the optical coatings. The results are given in Table 1 and show that the elastic modulus of the optical coating layer changes dramatically with a change in substrate. This demonstrates that the optical coating is structured such that flexing under an applied force, in this case an ultrafine diamond tip, occurs in the film such that the deformation matches that of the underlying substrate. Further analysis shows that the 150nm thick anti-reflective coating flexes to 5 microns before failure; that is the film deforms to 33 times its own thickness before failure occurs. The arrangement of the particles provides strength and flexibility by virtue of each particle having multiple contact points with surrounding particles.

This eliminates brittle failure and film delamination under impact and renders the film 25 suitable for outdoor applications such as solar cells and eyewear. For comparison, a typical hardcoated polycarbonate plaque has a hardness of 0.9 GPa and an elastic modulus of 9.0GPa. A typical anti-reflective coating deposited by physical vapour deposition (e.g. supplied by Norville) shows that brittle film delamination begins when the indenter penetrates to the thickness of the film, approximately 200nm, because there is no elastic 30 deformation possible in such layers.

Table 1.

Substrate	Film Hardness (GPa)	Film Elastic modulus (GPa)
Quartz	1.5	63.2
Silicone hardcoated polycarbonate	1.1	7.3

APPLICATIONS

5 The optical coating can be used numerous fields such as optics, ophthalmics, displays, solar collection, lighting components, windows for buildings, vehicle windows (e.g. automotive), glass for protecting pictures/paintings, display cases, fish tanks/aquaria, and instrument display panels. One exemplary application of the optical coating is on a glass or polymer window on top of a photovoltaic solar cell. The solar cell may be of any suitable kind, such as monocrystalline silicon, polycrystalline silicon, thin-film silicon and hybrid technologies.

10 The optical coating may be used on other optical components, known as solar concentrators, used for collecting and directing sun light to a photovoltaic cell. Suitable polymer materials for such components include, but are not limited to, polyesters such as polyethylene terephthalate (PET) and polyethylene naphthalate (PEN), and polyolefins such as biaxially oriented polypropylene (BOPP). However, the optical coating embodying the invention may

15 also be used in general displays, and general window applications – for example for thermal management of buildings. An optical coating embodying the invention can also be employed in ophthalmic elements, whether made of glass or plastics materials, for example spectacle lenses.

Claims

1. An optical coating comprising a binder and a plurality of porous silica nanoparticles in which the pores are randomly oriented, and wherein said pores have an internal surface at least partially comprising a hydrophobic polymer layer.
2. An optical coating according to claim 1, wherein the refractive index of the coating is in the range of from 1.0 to 1.4.
3. An optical coating according to claim 1 or 2, which is an antireflective coating.
4. An optical coating according to any one of the preceding claims, wherein the reflectance for incident light of at least one wavelength in the range from 300 nm to 1900 nm is less than 2%, preferably less than 1.5%.
5. An optical coating according to any one of the preceding claims, wherein the particle size of the porous silica nanoparticles is less than 50nm, most preferably in the range of from 20nm to 30nm
6. An optical coating according to any one of the preceding claims, wherein the particle internal pore surfaces comprise a hydrophobic polymer layer of up to 30 wt% of the particle mass.
7. An optical coating according to any one of the preceding claims, wherein the hydrophobic polymer layer comprises polystyrene or poly vinyl butadiene
8. An optical coating according to any one of the preceding claims wherein the porous silica nanoparticles are distributed within the binder.
9. An optical coating according to any one of the preceding claims wherein the binder comprises at least one of silicate, silica, silicone based polymer, siloxane based polymer, acrylate based polymer, cellulose, cellulose derivatives, and vinyl alcohol.
10. An optical coating according to any one of the preceding claims on a substrate, wherein the elastic modulus of the coating is greater than one half and less than twice the elastic modulus of the substrate.
11. An optical coating according to claim 10 in which the elastic modulus of the coating substantially matches the elastic modulus of the substrate.
12. An optical coating according to any one of the preceding claims provided on a substrate, wherein the substrate comprises at least one of glass, quartz, polycarbonate, silicone hardcoated polycarbonate, acrylate coated polycarbonate, polymethyl methacrylate, polyethylene terephthalate, polyethylene naphthalate, or cellulose triacetate.

13. An optical coating according to any one of the preceding claims, wherein the thickness of the coating is in the range of from 75nm to 500nm.
14. An optical coating according to any one of the preceding claims, wherein the porous silica nanoparticles comprise at least one of mesoporous particles and microporous particles.
- 5 15. A solar cell comprising an optical coating according to any one of the preceding claims.
16. A display comprising an optical coating according to any one of the preceding claims.
17. A lighting component comprising an optical coating according to any one of the preceding claims.
- 10 18. An ophthalmic element comprising an optical coating according to any one of the preceding claims.
19. A window comprising an optical coating according to any one of the preceding claims.
20. A method of fabricating an optical coating, said method comprising:
- 15 providing a blend of porous silica nanoparticles in which the pores are randomly oriented, and wherein said pores have an internal surface at least partially comprising a hydrophobic polymer layer, in conjunction with a binder and a solvent;
- coating the particles, binder and solvent as a layer onto a substrate; and
- removing solvent from the layer to form the optical coating.
- 20 21. A method according to claim 20, further comprising:
- curing the coating by maintaining the temperature in the range of from 50 to 250°C, more preferably from 80 to 140°C.
22. A method according to claim 21, wherein removing the solvent and curing the coating are performed simultaneously.
- 25 23. A method of fabricating an optical coating according to any one of claims 20 to 22 in which the solvent is primarily alcohol based.
24. A method of fabricating an optical coating according to any one of claims 20 to 23 in which the binder comprises at least one of silicate, silica, silicone based polymer, siloxane based polymer, acrylate based polymer, cellulose, cellulose derivatives, and vinyl alcohol.
- 30 25. A method of fabricating an optical coating according to any one of claims 20 to 24 in which the substrate comprises at least one of glass, quartz, polycarbonate, silicone hardcoated polycarbonate, acrylate coated polycarbonate, polymethyl methacrylate, polyethylene terephthalate, polyethylene naphthalate, or cellulose triacetate.

26. A method of fabricating an optical coating according to any one of claims 20 to 25 wherein the porous silica nanoparticles comprise at least one of mesoporous particles and microporous particles
27. A method of fabricating an optical coating according to any one of claims 20 to 26 in which the coating process comprises at least one of spin coating, dip coating, roll to roll coatings, web coating or spray coating.

ABSTRACT

OPTICAL COATING

- 5 The optical coating comprises a binder-nanoparticle composite film in which porous silica nanoparticles have randomly oriented pores. The internal surfaces of the pores are coated with a thin (e.g. monolayer) of polymer – typically polystyrene. The pores are primarily air filled except for this thin polymer internal surface. There is also a layer of polymer on the outer surface which acts to link the particles together when deposited in a thin film prior to
- 10 the majority of inter-particle space being filled with a binder. Due to the hydrophobic nature of the internal pore structure and its random nature there is substantially no binder ingress into the pores and the refractive index of the particles is maintained at less than 1.20. The binder material will typically have a refractive index of greater than 1.5. The refractive index of the mixture of the particles and the binder system can be tailored to a specific substrate.
- 15 This allows the optimization of the refractive index of the coating to minimise the reflectivity, for example for use as an anti-reflective coating.

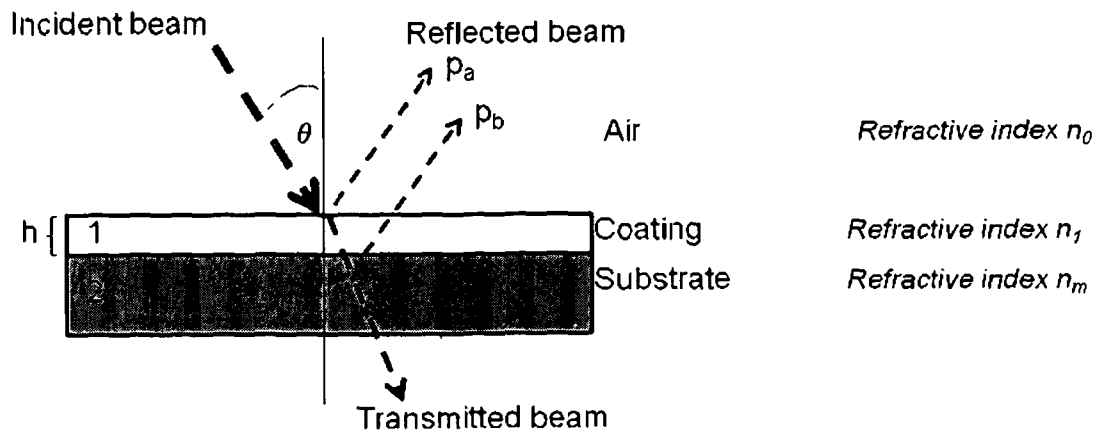


Fig. 1

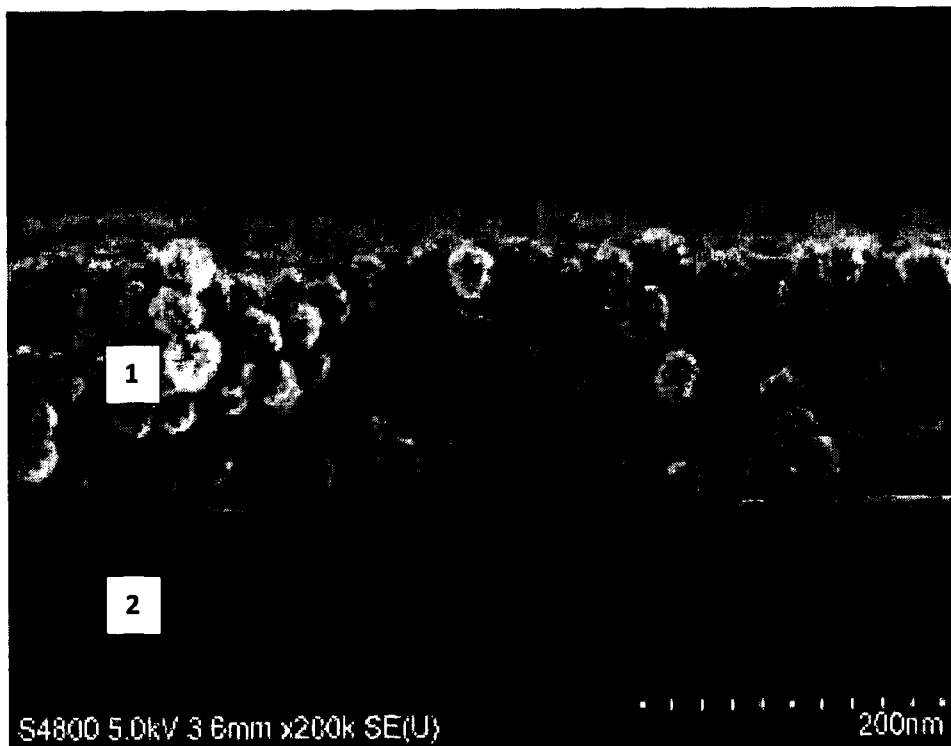


Fig. 2

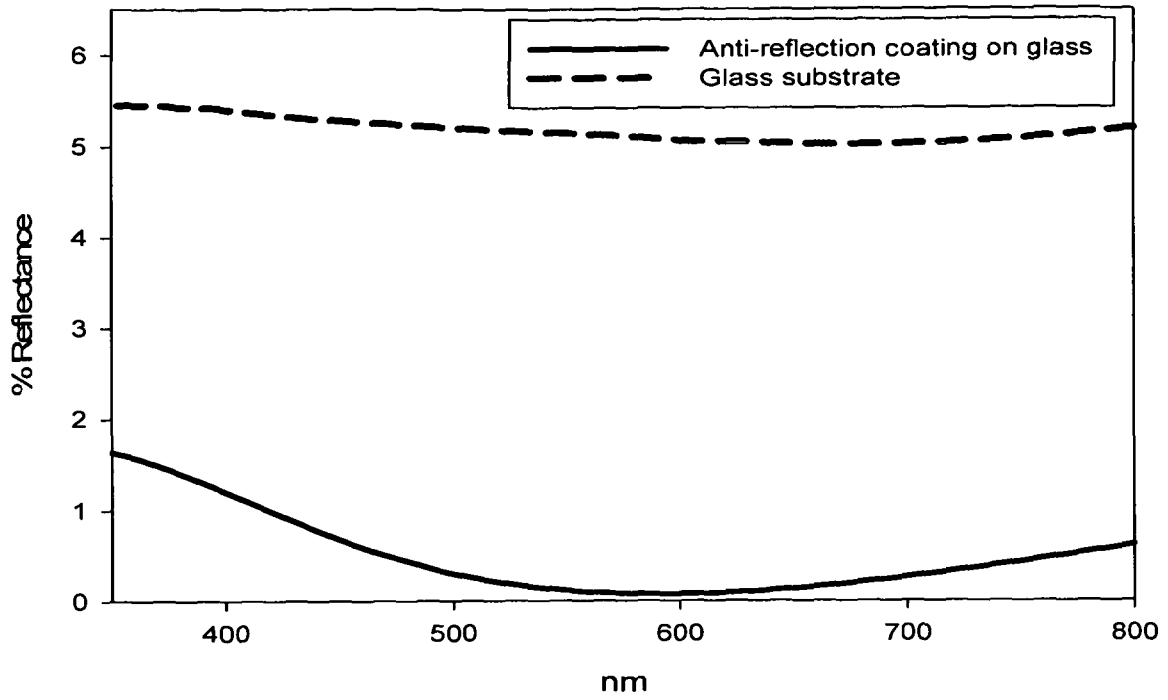


Fig 3



Fig. 4

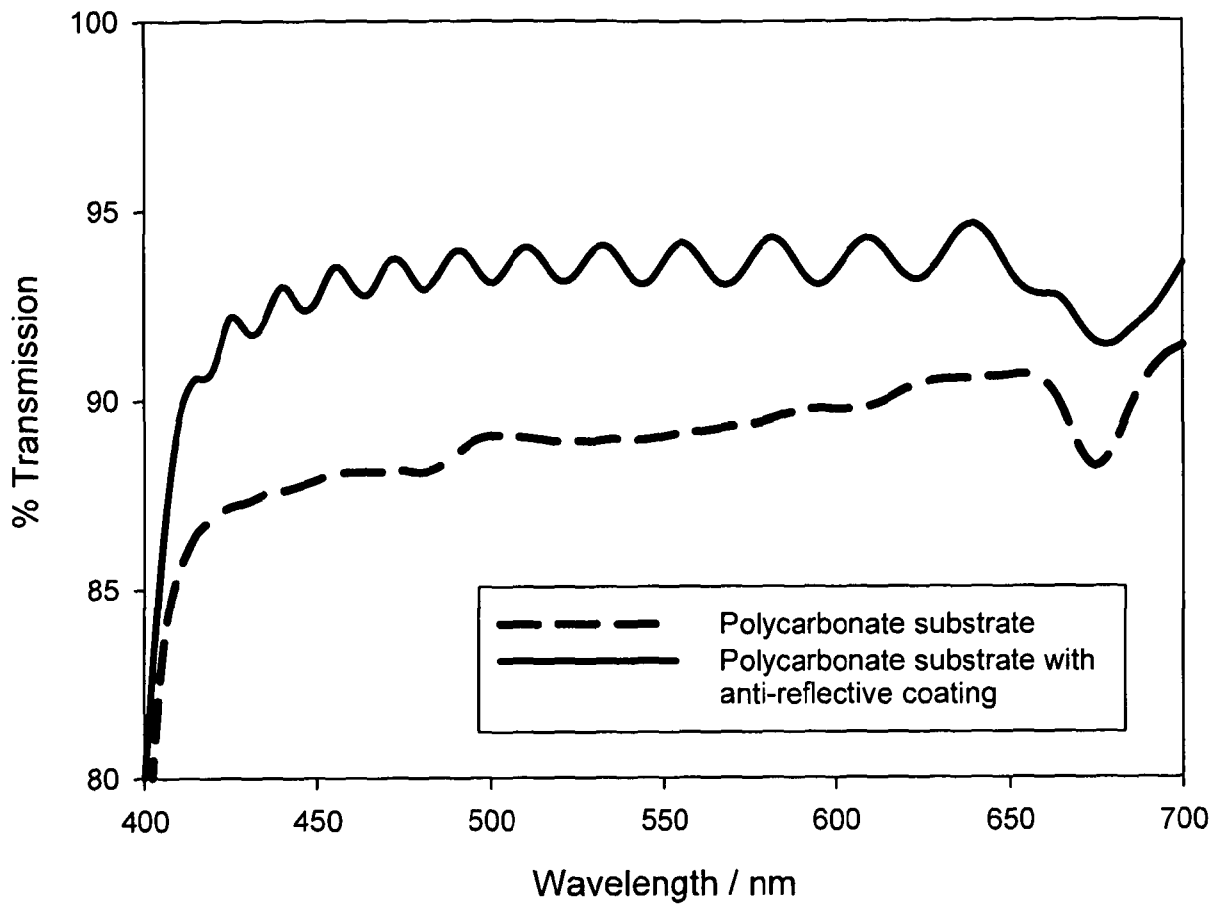


Fig. 5