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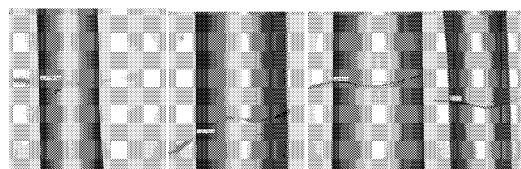
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(54) Title: UV-CURABLE DIELECTRIC INKS FOR A HYDROFLUORIC ACID MASK ON GLASS SUBSTRATES

LINE 1 LINE 2 LINE 3 LINE 4



Pre HF Exposure



Post HF Exposure

(57) Abstract: A composition for a uv-curable dielectric ink comprising a UV curable monomer and a photoinitiator for a hydrofluoric acid mask on glass substrates.

FIG. 1

UV-CURABLE DIELECTRIC INKS FOR A HYDROFLUORIC ACID MASK ON GLASS SUBSTRATES

BACKGROUND INFORMATION

5 Field of the Disclosure

The present invention relates to uv-curable dielectric inks for a hydrofluoric acid mask on glass substrates.

Description of the Related Art

Hydrofluoric acid (HF) etches by "eating" at the surface of exposed
10 glass. Over the past years, HF etching, has been used in various industries. Commonly, industrial companies dip stenciled glass into a tank of acid wherein the unexposed areas are protected by a stencil which creates the pattern on glass. This practice is less used today by glass artists and crafters, but is used in a wide variety of manufacturing glass
15 products. Today, it is used to etch glass by reacting with silicon dioxide to form gaseous or water-soluble silicon fluorides in a variety of industries including computer components. This reaction results in the removal of some of the silica from the surface of the glass object from the oxide matrix. To assure that the etching occurs only where it is desired, wax or
20 some other nonreactive substance is applied where etching is unwanted.

Conventional epoxy acrylates based on diglycidyl ether bisphenol A are the most frequently used oligomers in the ultraviolet light (UV) curing industry because they are hard, fast curing, and offer excellent abrasion and chemical resistance. Unfortunately, the inherent hardness of these
25 oligomers has several negative side effects, including yellowing over time, high viscosities and poor adhesion.

UV and/or visible light radiation is used to induce photochemical polymerization or crosslinking of a monomer, oligomer or prepolymer formulation containing a certain type of unsaturated group, such as an
30 acrylic group, and an appropriate initiator. The initiator is used to absorb the light energy and transform it into active species, such as radicals or ions, capable of inducing such reactions. Applications extend to general coatings for paper, board, wood, tapes, compact discs and holograms,

inks, photoresists for imaging processes and adhesives for welding and sealing in electronic circuit boards. The photoinitiator is the key to the control of these processes and, in recent years, has seen many new developments. These include the need for water-soluble, co-reactive and polymeric structures with low migration rates, as well as cheaper UV/visible sensitizers with enhanced speed.

SUMMARY

In a first embodiment the invention is directed to a composition for uv-curable dielectric inks for a hydrofluoric acid mask on glass substrates, including a UV-curable monomer and at least one photoinitiator.

In another embodiment, the invention is directed to a process for the preparation of a composition for uv-curable dielectric inks for a hydrofluoric acid mask on glass substrates. The process includes mixing a UV-curable monomer, at least one photoinitiator and dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity of about 5.0 Pascal. The UV-curable monomer is a bisphenol-A based epoxy acrylate.

In another embodiment, the invention is directed to an electronic material display glass, including a glass substrate and dielectric coating. The physical dimensions of the dielectric coating, upon exposure to 5% (percent) hydrofluoric acid bath at 30° Celsius for 15 minutes, are reduced by no more than 1% (percent).

In yet another embodiment, the invention is directed to a process for needle-printing a dielectric coating for a hydrofluoric acid mask onto a glass substrate including the steps of preparing a composition for uv-curable dielectric inks by mixing a UV-curable bisphenol-A based epoxy acrylate monomer, at least one photoinitiator and dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity of about 5.0 Pascal. The ratio of the at least one photoinitiator to the monomer is 1 to 10 and the ratio of the dissolving solution to monomer is 1 to 2. The composition is loaded into a needle-printing device and the composition is needle printed on a glass substrate.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a photograph of the composition before and after exposure to hydrofluoric acid.

DETAILED DESCRIPTION

In a first embodiment, the invention is directed to a composition for uv-curable dielectric inks for a hydrofluoric acid mask on glass substrates including a UV-curable monomer and at least one photoinitiator. In the present embodiment, the monomer is a bisphenol-A based epoxy acrylate.

10 The selection of bis-phenol-A based epoxy acrylate monomers as a base formulation provides good adhesion to glass substrates and also good dimension stability. These oligomers are commercially available by Sartomer® America, Exton, PA (USA).

Specific variations of the oligomers include various monomers with
15 both epoxy and bisphenol A. One skilled in the art would recognize blends or a separate class of epoxy or bisphenol A acrylate may work well in the present invention. Carefully selecting the monomer and oligomer product, appreciating their special chemical structures and properties, are required to achieve the desired properties.

20 At least one photoinitiator is required such as Irgacure 651, commercially available from BASF Corporation. Additional photoinitiators are commonly selected from the group of ethyl 4-dimethylaminobenzoate and isopropyl thioxanthone and act as assistant co-initiators to enhance performance. Other photo initiator packages (PIPs) are acceptable to
25 some extent, depending on requirements for photospeed, thickness etc. Suitable photoinitiation systems are those, which generate free radicals upon exposure to actinic light at ambient temperature. These include the substituted or unsubstituted polynuclear quinones which are compounds having two intracyclic carbon atoms in a conjugated carbocyclic ring
30 system, e.g., 2-benzyl-2- (dimethylamino)-1-(4-morpholinophenyl)-1-butanone, 2,2-dimethoxy-2-phenylacetophenone, 9,10-anthraquinone, 2-methylantraquinone, 2-ethylantraquinone, 2-tert-butylantraquinone, octamethylantraquinone, 1,4-naphthoquinone, 9,10-phenanthrenequinone, benz (a) anthracene-7, 12-dione,

2,3-naphthacene-5,12-dione, 2-methyl-1,4-naphthoquinone, 1,4-dimethyl-anthraquinone, 2,3-dimethylantraquinone, 2-phenylantraquinone, 2,3-diphenylantraquinone, retenequinone, 7,8,9,10-tetrahydronaphthracene-5,12-dione, and 1,2,3,4-tetra-
5 hydrobenz(a)anthracene-7,12-dione.

Other photoinitiators which are also useful, even though some may be thermally active at temperatures as low as 85°C, are described in U.S. Patent No. 2,760,863, herein incorporated by reference, and include vicinal ketaldonyl alcohols such as benzoin, pivaloin, acyloin ethers, e.g.,
10 benzoin methyl and ethyl ethers; α -hydrocarbon-substituted aromatic acyloins, including α -methylbenzoin, α -allylbenzoin and α -phenylbenzoin, thioxanthone and/or thioxanthone derivatives and the appropriate hydrogen donors. Photoreducible dyes and reducing agents disclosed in U.S. Patent Nos. 2,850,445, 2,875,047, 3,097,096, 3,074,974, 3,097,097,
15 and 3,145,104, herein incorporated by reference, as well as dyes of the phenazine, oxazine, and quinone classes, Michler's ketone, benzophenone, 2,4,5-triphenylimidazolyl dimers with hydrogen donors including leuco dyes and mixtures thereof as described in U.S. Patent Nos. 3,427,161, 3,479,185, and 3,549,367, herein incorporated by
20 reference, can be used as initiators. Also useful with photoinitiators and photoinhibitors are sensitizers disclosed in U.S. Patent No. 4,162,162, herein incorporated by reference. The photoinitiator or photoinitiator system is present in 0.05 to 10 percent by weight, based on the total weight of a dry photopolymerizable layer.

25 The composition further comprises a dissolving component to adjust and control viscosity. The formulations for the compositions of the present invention are primarily designed for draw-down coating and screen-printing. As would be appreciated by one skilled in the art, viscosity of the formulations is about 5 Pascal (Pas). For needle-printing,
30 about 5% more dissolving solution will be added to make viscosity below 0.5 Pas. Most commonly, the dissolving component of the present invention is methyl ethyl ketone (MEK). Other solvents such as iso-propyl alcohol (IPA), acetone and other common solvents can be used. Solvents

are removed before UV-curing. The amount of solvent can vary based on the viscosity desired.

The composition may further include a silyl acrylate monomer. Addition of silyl acrylate monomers enables further linking of UV-cured mask film to glass surface by reaction of silicon atoms of acrylate monomers to silicon atoms of glass surface, upon HF catalysis. Silicon atoms in coating layer can react with silicon atoms to form silicon-oxygen-silicon (Si-O-Si) bonds under catalysis of a strong acid, such as HF to further enhance adhesion of the coating with the substrate.

Without limiting the invention, the silyl acrylate monomer is commonly tri(methoxyl)propyl acrylate, but any monomer which can further enhance linking of UV-cured mask film to glass surface by reaction of silicon atoms of acrylate monomers to silicon atoms of glass surface, upon HF catalysis is acceptable.

In its most basic form, limited to the monomer and photoinitiator, the composition has a formulation wherein the monomer is greater than 90 percent by weight of the composition. The addition of the dissolving solution and/or the silyl acrylate monomer redefines the composition wherein greater than 70 percent by weight of the composition is the bis-phenol-A based epoxy acrylate monomers. However, the ratio of photoinitiator to bis-phenol-A based epoxy acrylate monomer of 1 to 10 is preferred to ensure the functional aspects of the invention. However, this ratio can be expanded to 1 to 20. Further, as discussed, the viscosity of the composition is commonly 5 Pascal, however, one skilled in the art would recognize modifying the amount of dissolving solution would change the viscosity as desired and required for various printing techniques. The ratio of monomer(s) to PIPs is important. However, in the present invention, since formulations do not contain fillers and are transparent, the ratio variation of PIP to monomer could exceed 1 to 10 and be acceptable. The ratio of monomers and PIPs to solvent does not matter for the final UV-curing and adhesion performance because solvents are removed before UV-curing and HF treatment.

In another embodiment, the invention is directed to a process for the preparation of a composition for uv-curable dielectric inks for a

hydrofluoric acid mask on glass substrates. The process includes the steps of mixing a UV-curable monomer, at least one photoinitiator and a dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity
5 of about 5.0 Pascal. The UV-curable monomer is a bisphenol-A based epoxy acrylate.

In a preferred mode of the present embodiment, as provided in Example 1 herein and for illustration of ratios, the mixture is 15.3 grams UV-curable monomer (CN110; difunctional bisphenol A-based epoxy
10 acrylate oligomer epoxy acrylate from Sartomer® (USA), 0.42 grams photoinitiator, 0.36 grams, ethyl 4-dimethylaminobenzoate, 0.36 grams isopropyl thioxanthone and 3.6 g dissolving solution; the dissolving solution is methyl ethyl ketone

In another embodiment, the invention is directed to an electronic
15 material display glass comprising a glass substrate and dielectric coating wherein the physical dimensions of the dielectric coating upon exposure to 5% (percent) hydrofluoric acid bath at 30 Celsius for 15 minutes are reduced by no more than 1% (percent). The dielectric coating includes a UV-curable monomer of a bisphenol-A based epoxy acrylate, at least on
20 photoinitiator and a dissolving solution. In the present embodiment, the ratio of the at least one photoinitiator to bisphenol-A based epoxy acrylate monomer is 1 to 10 and the ratio of the dissolving solution to bisphenol-A based epoxy acrylate monomer is 1 to 5; the dissolving solution is methyl ethyl ketone.

25 As illustrated in FIG. 1, the composition of Example 1 of the present invention was printed on a glass substrate and UV cured to become solid lines. The widths of the lines were measured to provide a pre HF exposure measurement result as provided in TABLE 1. The glass substrates were immersed into a 5% HF bath at 30° C for 15 minutes, then rinsed by water
30 and air-dried. After such a process, line width was measured again to obtain the post HF exposure result. As provided by TABLE 1, the difference in the pre and post exposure to HF is less than 1 percent.

TABLE 1

	LINE 1	LINE 2	LINE 3	LINE 4
Pre HF	1096.7 μ m	1915.7 μ m	2053.9 μ m	1944.8 μ m
Post HF	1088.4 μ m	1906.1 μ m	2066.3 μ m	1948.9 μ m

5

In yet another embodiment, the invention is directed to a process for needle-printing a dielectric coating for a hydrofluoric acid mask onto a glass substrate. The process includes the steps of preparing a composition for UV-curable dielectric inks by mixing a UV-curable

10 bisphenol-A based epoxy acrylate monomer, at least one photoinitiator and dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity of about 5.0 Pascal. For the composition, the ratio of at least one photoinitiator to the monomer is 1 to 10 and the ratio of dissolving solution

15 to monomer is 1 to 2. The composition is input into a needle-printing device. The composition is needle printed onto a glass substrate. Adjusting the viscosity of the composition for needle printing or similar printing technique, allows the ability to specifically contact the glass substrate location(s) as required.

20

EXAMPLES

The concepts described herein will be further described in the following examples, which do not limit the scope of the invention described in the claims.

Example 1

25 A mixture of 15.3 grams UV-curable monomer, (CN110, from Sartomer®), 0.42 grams photoinitiator, Irgacure 651, from BASF, 0.36 grams, ethyl 4-dimethylaminobenzoate or Quantacure EPD from Quantacure, 0.36 grams isopropyl thioxanthone or Quatacure ITX from Quatacure and 3.6 g methyl ethyl ketone from Sigma-Aldrich was well

30 mixed by a mechanical stirrer at room temperature for 2 hours, resulting in a transparent pale yellow color liquid. This liquid was coated by draw-

down coating on glass substrate. Such coated substrates were dried at 80° C for 10 minutes, and exposed to UV-light from a high pressure mercury lamp at 220 mm/cm², resulting in solid coating about 10 microns thick. Such obtained UV-cured parts were put into a 5% hydrofluoric acid bath at
5 30° C for 15 minutes. No “peeling” of the UV-cured film from the glass surface was observed using the transparent adhesive tape test (Tape Test). The Tape Test is widely used in the industry to detect “peeling” of coatings from surfaces.

Example 2

10 The liquid in Example 1 was diluted by methyl ethyl ketone (MEK) by 100% (10 g above liquid and 10 g MEK). The diluted liquid was printed by spring needles on the same glass substrates, resulting in 50 to 500 microns wide lines, depending on printing speed. Then such printed parts were dried, UV-cured, HF treated and peeling tested in the same way as
15 in Example 1. No peeling was observed using the Tape Test.

Example 3

A mixture of 20 g liquid in Example 2 and 5 grams of tri(methoxyl)propyl acrylate from Sigma-Aldrich was mixed at room temperature by hand for 10 minutes. Resulting liquid was processed the
20 same way as in Example 2. No peeling was observed after HF treatment by the Tape Test.

25

CLAIMS

What is claimed is:

1. A composition for uv-curable dielectric inks for a hydrofluoric acid
5 mask on glass substrates comprising:
 - a. UV-curable monomer; and
 - b. at least one photoinitiator.
2. The composition of claim 1, wherein the monomer is a bisphenol-A based epoxy acrylate.
- 10 3. The composition of claim 2, wherein the at least on photoinitiator is selected from the group consisting of ethyl 4-dimethylaminobenzoate and isopropyl thioxanthone.
4. The composition of claim 3, wherein the composition further comprises a dissolving component.
- 15 5. The composition of claim 4, wherein the dissolving component is methyl ethyl ketone.
6. The composition of claim 5, wherein the composition further comprises a silyl acrylate monomer.
7. The composition of claim 6, wherein the silyl acrylate monomer is
20 tri(methoxyl)propyl acrylate.
8. The composition of claim 1, wherein the monomer is greater than 90 percent by weight of the composition.
9. The composition of claim 4 or 5, wherein the composition is greater than 70 percent by weight of the composition.
- 25 10. The composition of claim 3, wherein the ratio of photoinitiator to monomer is 1 to 10.
11. The composition of claim 10, wherein the viscosity of the composition is 5 Pascal.
12. A process for the preparation of a composition for uv-curable
30 dielectric inks for a hydrofluoric acid mask on glass substrates comprising:
 - a. mixing a UV-curable monomer at least one photoinitiator and dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity of about 5.0 Pascal,

wherein the UV-curable monomer is a bisphenol-A based epoxy acrylate.

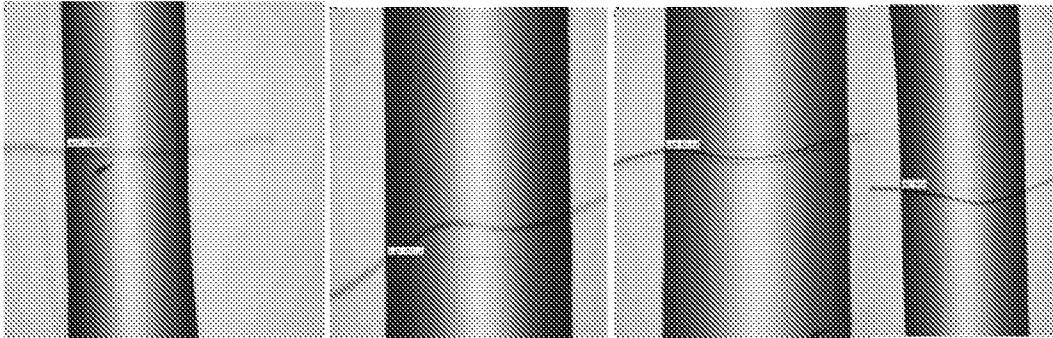
13. The process of claim 12, wherein the mixture is of 15.3 grams UV-curable monomer, 0.42 grams photoinitiator, 0.36 grams, ethyl 4-dimethylaminobenzoate, 0.36 grams isopropyl thioxanthone and 3.6 g dissolving solution,

wherein the dissolving solution is methyl ethyl ketone.

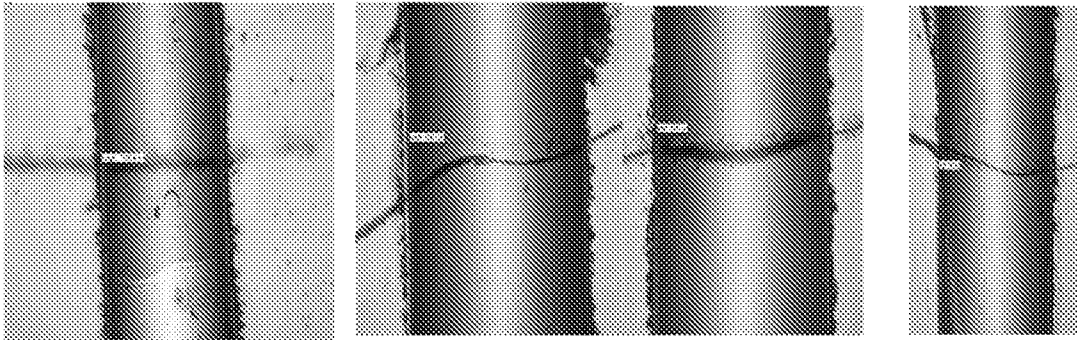
14. A composition for uv-curable dielectric inks for a hydrofluoric acid mask on glass substrates made by the process of claim 13.
15. An electronic material display glass comprising a glass substrate and dielectric coating wherein the physical dimensions of the dielectric coating upon exposure to 5% (percent) hydrofluoric acid bath at 30 Celsius for 15 minutes are reduced by no more than 1% (percent).
16. The electronic material display glass of claim 15, wherein the dielectric coating comprises a UV-curable monomer of a bisphenol-A based epoxy acrylate, at least one photoinitiator and a dissolving solution.
17. The electronic material display glass of claim 16, wherein the ratio of the at least one photoinitiator to bisphenol-A based epoxy acrylate monomer is 1 to 10.
18. The electronic material display glass of claim 17, wherein the ratio of the dissolving solution to bisphenol-A based epoxy acrylate monomer is 1 to 5.
19. The electronic material display glass of claim 18, wherein dissolving solution is methyl ethyl ketone.
20. A process for needle-printing a dielectric coating for a hydrofluoric acid mask onto a glass substrate comprising the steps of:
- preparing a composition for uv-curable dielectric inks by mixing a UV-curable bisphenol-A based epoxy acrylate monomer, at least one photoinitiator and dissolving solution by a mechanical stirrer at room temperature for about 2.0 hours, resulting in a transparent pale yellow color liquid with a viscosity of about 5.0 Pascal, wherein the ratio of at least one photoinitiator to the monomer is 1 to 10 and the ratio of dissolving solution to monomer is 1 to 2;
 - loading the composition into a needle-printing device; and

- c. needle printing the composition on a glass substrate.

LINE 1	LINE 2	LINE 3	LINE 4
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Pre HF Exposure



Post HF Exposure

FIG. 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2015/041297

A. CLASSIFICATION OF SUBJECT MATTER

INV. B41M3/00 C03C15/00 C09D4/00 C09D11/00 C09D11/101
ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

B41M C03C C09D

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

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

EP0-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2011/033664 A1 (DICKER MARK ROBERT [GB] ET AL) 10 February 2011 (2011-02-10)	1-4,6-8, 10-14, 16-19 20
Y	paragraphs [0114], [0115], [0175] - [0184]; claims 1,5; examples 1-4 -----	
X	WO 2004/009651 A1 (CIBA SC HOLDING AG [CH]; HUESLER RINALDO [CH]; FUCHS ANDRE [DE]) 29 January 2004 (2004-01-29)	1-8, 10-19
Y	page 38, line 1 - page 42, line 35; claims 1-8	20
A	-----	9
X	US 2004/209202 A1 (NALEWAJEK DAVID [US] ET AL) 21 October 2004 (2004-10-21)	1,2,15
Y	paragraphs [0071], [0072], [0085], [0086], [0122]; claims 1,3,4,7,10 ----- -/--	20



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International application No
PCT/US2015/041297

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP 0 263 990 A1 (CARIBONUM LTD [GB]) 20 April 1988 (1988-04-20) claim 1; example 1 -----	20

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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