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

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
13 February 2003 (13.02.2003)

PCT

(10) International Publication Number
WO 03/012162 A1

(51) International Patent Classification^7: **C23C 16/44**, 16/458, C25F 3/04

(21) International Application Number: PCT/US02/23287

(22) International Filing Date: 22 July 2002 (22.07.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/918,683 27 July 2001 (27.07.2001) US

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(81) Designated States *(national)*: CN, JP, KR, SG.

(84) Designated States *(regional)*: European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR).

Published:
— *with international search report*

(54) Title: ELECTROCHEMICALLY ROUGHENED ALUMINUM SEMICONDUCTOR PROCESSING APPARATUS SURFACES

(57) Abstract: A uniform, controllable method for electrochemically roughening an aluminum-comprising surface to be used in a semiconductor processing apparatus is disclosed. Typically the aluminum-comprising surface is aluminum or an aluminum alloy. The method involves immersing an aluminum-comprising surface in an HCl solution having a concentration ranging from about 1 volume % to about 5 volume %, at a temperature within the range of about 45 °C to about 80 °C, then applying an electrical charge having a charge density ranging from about 80 amps/ft.^{2} to about 250 amps/ft.^{2} for a time period ranging from about 4 minutes to about 25 minutes. A chelating agent may be added to enhance the roughening process. The electrochemical roughening method can be used on aluminum alloys in general, including but not limited to 6061 and LP. The electrochemical roughening provides a smoothly rolling surface which does not entrap particles and which provides increased surface area for semiconductor process byproduct adhesion. The roughened surface provides an excellent surface for subsequent anodization.

[0001] ELECTROCHEMICALLY ROUGHENED ALUMINUM SEMICONDUCTOR PROCESSING APPARATUS SURFACES

[0002] BACKGROUND OF THE INVENTION

[0003] 1. Field of the Invention

[0004] The present invention pertains to an electrochemically roughened aluminum surface for use within a semiconductor processing chamber. The present invention also pertains to a method of electrochemically roughening an aluminum surface. The roughened surface is typically anodized to provide a finished surface for use in semiconductor processing.

[0005] 2. Brief Description of the Background Art

[0006] Semiconductor manufacturing processes, such as etch and deposition processes, utilize a wide variety of processing gases and substrate materials. Highly volatile process byproducts are typically removed from the processing chamber by application of vacuum. Less volatile byproducts may adhere to the interior surface of the processing chamber or may redeposit on the surface of the semiconductor substrate being processed. Most semiconductor manufacturers prefer to have redepositing byproducts deposit on processing chamber surfaces (rather than the substrate). The processing chamber surfaces are then periodically cleaned. Frequent chamber cleanings are expensive in terms of processing chamber downtime. The more redeposited byproducts which can be held by the processing chamber surfaces, the less frequent the cleaning requirement.

[0007] Interior surfaces of semiconductor processing chambers are frequently aluminum. One prior art semiconductor processing chamber includes anodized aluminum surfaces which have been lapped to have a surface roughness of only 4 Ra, which is essentially a mirror finish. However, when subjected to the high temperatures and processing conditions used in many semiconductor manufacturing processes, the highly polished, anodized aluminum surface developed numerous tiny cracks in the anodized layer, known as craze lines; these are shown in Figure 1. While the craze lines 100 typically do not penetrate all of the way through the anodized layer to the boundary layer at the base aluminum beneath, they tend to spread across the anodized surface, producing a spider web pattern. During a fluorine-based etch process, the anodized aluminum surface reacts with fluorine gas, causing the craze lines to fill with a self-passivating fluoride. Although the craze lines may not interfere with the operation of the chamber during a fluorine-based etch process, they are cosmetically unappealing, and the user of the processing chamber tends to worry that fluorine-containing species may be passing through the protective anodized layer and corroding the aluminum surface beneath. Further, in a non-fluorine-based environment (such as during a chlorine-based etch process), the craze lines do not fill with self-passivating fluoride and the anodized surface may eventually fail, exposing the aluminum beneath to corrosion by chlorine-containing species.

[0008] During a number of semiconductor processing procedures, byproducts are formed which are not sufficiently volatile to be removed by the vacuum system of the processing chamber. In many instances, it is desirable to provide a surface inside the processing chamber on which these byproducts are capable of adhering, so that they will not fall upon semiconductor workpieces during processing, causing contamination.

[0009] One method of improving the adhesion of semiconductor processing byproducts to an aluminum surface within a semiconductor processing chamber is to provide a roughened surface to which byproducts generated during processing can stick. Typically, aluminum semiconductor chamber surfaces have been roughened by bead blasting. However, bead blasting often is a manual process, in which it is difficult to control the uniformity and repeatability. Further, bead blasting typically provides a very sharp, jagged surface 200 on the aluminum, as shown in Figure 2. Tips of the roughened aluminum can curl over, forming hook-shaped projections 202 which can break off or entrap particles 204, including the bead blast particle itself. As a result, the bead blasting media may act as a source of contamination of the aluminum surface. Bead blasting is not useful as a roughening method for some of the softer aluminum alloys, such as the 1000 series, because the bead blasting particles can easily become embedded in the ductile metal. Further, the sharp surface provided by bead blasting may complicate a subsequent anodization process.

[0010] It would therefore be desirable to provide a uniform and controllable method for roughening an aluminum surface which could be used for all aluminum alloys. In particular, the roughening method should provide a surface which does not entrap particles, is free from jagged and hooked surface formations, and is easily anodized.

[0011] SUMMARY OF THE INVENTION

[0012] Applicants have discovered a uniform, controllable method for electrochemically roughening an aluminum-comprising surface intended for use within a semiconductor processing chamber. Typically the aluminum-comprising surface is aluminum or an aluminum alloy. Applicants have also determined that if they electrochemically roughen an aluminum or aluminum alloy surface, they avoid the formation of jagged and hooked surface topography. The surface which is formed by the electrochemical roughening provides a topography which resembles small rolling hills and valleys. The estimated average height of the hills above the valleys is approximately 16 μm; the estimated average distance between the hills is approximately 50 μm, depending on the grade of the aluminum. Typically, the height of the hills ranges from about 8 μm to about 25 μm, and the distance between the center of one hill and that of an adjacent hill ranges from about 30 μm to about 100 μm.

[0013] Surprisingly, the hill and valley topography obtained by electrochemically roughening an aluminum or aluminum alloy surface relieves stress in an anodized finish subsequently produced over the roughened surface, so that the anodized layer does not crack upon thermal cycling up to about 300°C. In addition, unexpectedly, the amount of redepositing byproduct which can be accumulated over the hills and valleys (including an anodized surface which mirrors the underlying aluminum surface) is drastically increased over that which can be accumulated over a bead-blasted surface. As a result, the number of substrate processing cycles prior to cleaning with the new, electrochemically roughened, aluminum or aluminum alloy anodized surface is about 5 times greater than with the bead blasted aluminum anodized surface. [0014]

Applicants' method for surface roughening can be used on aluminum and aluminum alloys in general, including but not limited to 6061 and LP (available from Alcan Alusuisse). Applicants' method promotes formation of a smooth, rolling-hilled, anodized surface which does not entrap particles. Further, applicants' electrochemically roughened aluminum-comprising surfaces provide increased surface area for collection of redepositing byproducts.

[0015] BRIEF DESCRIPTION OF THE DRAWINGS

[0016] Figure 1 shows a prior art anodized aluminum surface 100 which has been lapped to have a surface roughness of 4 Ra. Note the many craze lines 102 which have formed in the aluminum surface subsequent to exposure to process conditions, producing a spider web pattern.

[0017] Figure 2 shows a prior art aluminum surface 200 which has been roughened using bead blasting. Note the many hook-shaped projections 202 which can break off or entrap particles 204, including the bead blast particle itself.

[0018] Figure 3 shows an aluminum surface 300 which has been roughened using applicants' electrochemical roughening method. Note the smooth, rolling topography of applicants' electrochemically roughened aluminum surface.

[0019] DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

[0020] Applicants' invention pertains to a method of electrochemically roughening an aluminum-comprising surface. Typically the aluminum-comprising surface is aluminum or an aluminum alloy. Aluminum is commonly alloyed with elements such as silicon, copper, zinc, magnesium, manganese, iron, titanium, and nickel, by way of example, and not by way of limitation. Applicants' invention has use in semiconductor processing chambers which include electrochemically roughened aluminum surfaces, and particularly roughened surfaces having a protective coating thereover, such as an anodized aluminum coating. [0021] Applicants' method for electrochemically roughening an aluminum-comprising surface comprises immersing the aluminum-comprising surface in an aqueous HCl solution having a concentration ranging from about 1 volume % to about 5 volume % at a temperature ranging from about 45°C to about 80°C, then applying an electrical charge having a charge density ranging from about 80 amps/ft.^{2} to about 250 amps/ft.^{2} for a time period ranging from about 5 minutes to about 25 minutes. Chelating agents (such as, for example, but without limitation, gluconic acid, available from VWR Scientific Products, West Chester, PA) may be added to the HCl solution to control the bath chemistry and conductivity.

[0022] Typical processing conditions for electrochemically roughening aluminum and aluminum alloys according to applicants' method are presented in Table One, below.

[0023] Table One. Typical Process Conditions for Electrochemically Roughening Aluminum and Aluminum Alloys

Process Parameter	Typical Process Conditions	Preferred Process Conditions	Optimum Known Process Conditions
HCl Concentration (% volume)	1 - 5	1 - 3	1 - 1.5
Chelating Agent (% volume)	0.5 - 3	0.5 - 1.5	0.8 - 1.2
Tank Temperature (°C)	45 - 80	50 - 70	55 - 65
AC Frequency (Hz)	60 - 120	80 - 100	85 - 95
Charge Density (amps/ft.^{2})	80 - 250	120 - 250	150 - 250
Time (min.)	4 - 25	4 - 20	4 - 20

[0024] Processing conditions will need to be adjusted depending on the specific chemical composition of the particular aluminum alloy being roughened. Applicants have performed electrochemical roughening of several commercially available aluminum alloys. Specific processing conditions used during the electrochemical roughening of these alloys are presented in Table Two, below.

[0025] Table Two. Process Conditions for Electrochemically Roughening Particular Aluminum Alloys

Alloy	6061*	LP**
Process Condition		
HCl Concentration (% volume)	1.0 - 1.5	1.0 - 1.5
Gluconic Acid*** (% volume) (Chelating Agent)	0.9 - 1.1	0.9 - 1.1
Tank Temperature (°C)	55 - 65	55 - 65
AC Frequency (Hz)	85 - 95	85 - 95
Charge Density (amps/ft.^{2})	175 - 250	175 - 250
Time (min.)	6 - 12	4 - 8

* Can be obtained from any of the major aluminum manufacturers, such as Alcoa (Pittsburgh, PA), Alcan, Inc. (Montreal, Canada), and Reynolds Aluminum Supply Co. (Richmond, VA).

** Obtained from Alcan Alusuisse (Stegen, Germany).

*** Obtained from VWR Scientific Products (West Chester, PA).

[0026] Unroughened, machined aluminum and aluminum alloy typically has a surface roughness ranging from about 12 Ra to about 32 Ra. After performing applicants' electrochemical roughening method, the aluminum or aluminum alloy surface typically has a surface roughness ranging from about 100 Ra to about 200 Ra, preferably ranging from about 110 Ra to about 160 Ra.

[0027] As shown in Figure 3, applicants' aluminum and aluminum alloy roughening method provides a surface 300 having a topography resembling small rolling hills 302 and valleys 304. The estimated average height of the hills 302 above the valleys 304 is approximately 16 μm; the estimated average distance between the hills 302 is approximately 50 μm, depending on the grade of the aluminum. Typically, the height of the hills 302 ranges from about 8 μm to about 25 μm, and the distance between the center of one hill and that of an adjacent hill ranges from about 30 μm to about 100 μm. Applicants' electrochemically roughened aluminum or aluminum alloy surface provides increased surface area for collection of redepositing byproducts, but does not entrap particles.

[0028] Applicants' electrochemical roughening method is particularly useful for roughening aluminum and aluminum alloy surfaces which are subsequently protected by a plasma-resistant coating, for use within semiconductor processing chambers, such as an etch chamber or a deposition chamber. Applicants' method is particularly useful for roughening any apparatus surface which comes into contact with semiconductor processing byproducts. Applicants' electrochemically roughened aluminum or aluminum alloy surface provides pockets in the hills and valleys which provide for the accumulation of semiconductor processing byproducts, such as etch byproducts or CVD deposition byproducts, preventing the byproducts from redepositing on the surface of the semiconductor substrate being processed. It is helpful to use a protective coating applied over the aluminum ro aluminum alloy surface which provides for adhesion of depositing byproducts. Example protective coatings include anodic oxide, flame spray-deposited aluminum oxide, and other ceramic coatings which may be conductive or non-conductive.

[0029] In particular, during a fluorine-based etch process, fluorine and carbon from the etch process react to form a polymer which easily adheres to an electrochemically roughened, anodized aluminum surface.

[0030] Applicants' electrochemically roughened, anodized aluminum or anodized aluminum alloy surfaces can be included in etch chambers which are used for etching dielectric materials (including inorganic dielectric materials, such as silicon oxide, silicon nitride, silicon oxynitride, and tantalum pentoxide, and organic dielectric materials, such as an organic low-k dielectric material), metals (such as aluminum, copper, titanium, tantalum, and tungsten), and polysilicon, by way of example, and not by way of limitation.

[0031] Applicants' method can be used to create roughened surfaces for semiconductor processing chamber components such as wall liners, cathode liners, slit valve doors, slit valve liners, buffer inserts, and gas distribution plates, by way of example, and not by way of limitation.

[0032] Anodization of applicants' electrochemically roughened aluminum and aluminum alloy surfaces can be performed using conventional aluminum anodization techniques known in the art, such as by following Mil Standard No. A-8625F, by way of example, and not by way of limitation. Because applicants' roughening method relieves stress within the aluminum or aluminum alloy surface, the resulting anodized surface does not form craze lines, even when subjected to the temperature cycling which occurs due to particular semiconductor manufacturing processes. [0033]

Other protective, plasma-resistant coatings, such as flame spray-deposited aluminum oxide and other ceramic coatings, can be deposited or applied over a roughened aluminum or aluminum alloy surface using techniques known in the art. Ceramic coatings, either conductive or non-conductive, may be applied over a roughened, anodized surface.

[0034] The above described preferred embodiments are not intended to limit the scope of the present invention, as one skilled in the art can, in view of the present disclosure expand such embodiments to correspond with the subject matter of the invention claimed below.

CLAIMS

We claim:

1. A semiconductor processing chamber having at least one interior surface comprising electrochemically roughened aluminum or aluminum alloy.

2. The semiconductor processing chamber of Claim 1, wherein said at least one interior surface has a surface roughness ranging from about 100 Ra to about 200 Ra.

3. The semiconductor processing chamber of Claim 2, wherein said surface roughness ranges from about 110 Ra to about 160 Ra.

4. The semiconductor processing chamber of Claim 1, wherein said electrochemically roughened aluminum or aluminum alloy surface has the appearance of rolling hills and valleys, when magnified.

5. The semiconductor processing chamber of Claim 4, wherein the height of said hills ranges from about 8 μm to about 25 μm.

6. The semiconductor processing chamber of Claim 4 or Claim 5, wherein the distance between the center of one hill and the center of an adjacent hill ranges from about 30 μm to about 100 μm.

7. The semiconductor processing chamber of Claim 1, wherein said electrochemically roughened aluminum or aluminum alloy surface underlies a coating selected from the group consisting of an anodized coating, a flame spray-deposited aluminum oxide coating, a ceramic coating, and an anodized coating having a ceramic coating applied thereover.

8. The semiconductor processing chamber of Claim 1, wherein byproducts generated during an etch process or a deposition process adhere to said electrochemically roughened aluminum surface.

9. The semiconductor processing chamber of Claim 1, wherein said semiconductor processing chamber is selected from the group consisting of an etch chamber and a deposition chamber.

10. The semiconductor processing chamber of Claim 9, wherein said semiconductor processing chamber is an etch chamber which is used for etching a material selected from the group consisting of a dielectric material, a metal, and polysilicon.

11. The semiconductor processing chamber of Claim 9, wherein said semiconductor processing chamber is an etch chamber, and wherein fluorine and carbon from an etch process react to form a polymer which adheres to said electrochemically roughened aluminum surface.

12. A processing component for use within a semiconductor processing chamber, wherein said processing component has at least one electrochemically roughened aluminum or aluminum alloy surface.

13. The processing component of Claim 12, wherein said electrochemically roughened aluminum or aluminum alloy surface has a surface roughness ranging from about 100 Ra to about 200 Ra.

14. The processing component of Claim 13, wherein said surface roughness ranges from about 110 Ra to about 160 Ra.

15. The processing component of Claim 12, wherein said electrochemically roughened aluminum or aluminum alloy surface has the appearance of rolling hills and valleys, when magnified.

16. The processing component of Claim 15, wherein the height of said hills ranges from about 8 μm to about 25 μm.

17. The processing component of Claim 15 or Claim 16, wherein the distance between the center of one hill and the center of an adjacent hill ranges from about 30 μm to about 100 μm.

18. The processing component of Claim 12, wherein said electrochemically roughened aluminum or aluminum alloy surface underlies a coating selected from the group consisting of an anodized coating, a flame spray-deposited aluminum oxide coating, a ceramic coating, and an anodized coating having a ceramic coating applied thereover.

19. The processing component of Claim 12, wherein byproducts generated during an etch process or a deposition process adhere to said electrochemically roughened aluminum or aluminum alloy surface.

20. The processing component of Claim 12, wherein said processing component is used within a semiconductor processing chamber selected from the group consisting of an etch chamber and a deposition chamber.

21. The processing component of Claim 20, wherein said semiconductor processing chamber is an etch chamber which is used for etching a material selected from the group consisting of a dielectric material, a metal, and polysilicon.

22. The processing component of Claim 20, wherein said semiconductor processing chamber is an etch chamber, and wherein fluorine and carbon from an etch process react to form a polymer which adheres to said electrochemically roughened surface.

23. The processing component of Claim 12, wherein said processing component is selected from the group consisting of: a wall liner, a cathode liner, a slit valve door, a slit valve liner, a buffer insert, and a gas distribution plate.

24. A semiconductor processing apparatus surface, wherein said surface comprises electrochemically roughened aluminum or aluminum alloy.

25. The semiconductor processing apparatus surface of Claim 24, wherein said surface has a surface roughness ranging from about 100 Ra to about 200 Ra.

26. The semiconductor processing apparatus surface of Claim 25, wherein said surface roughness ranges from about 110 Ra to about 160 Ra.

27. The semiconductor processing apparatus surface of Claim 24, wherein said electrochemically roughened aluminum or aluminum alloy surface has the appearance of rolling hills and valleys, when magnified.

28. The semiconductor processing apparatus surface of Claim 27, wherein the height of said hills ranges from about 8 μm to about 25 μm.

29. The semiconductor processing apparatus surface of Claim 27 or Claim 28, wherein the distance between the center of one hill and the center of an adjacent hill ranges from about 30 μm to about 100 μm.

30. The semiconductor processing apparatus surface of Claim 24, wherein said surface underlies a coating selected from the group consisting of an anodized coating, a flame spray-deposited aluminum oxide coating, a ceramic coating, and an anodized coating having a ceramic coating applied thereover.

31. The semiconductor processing apparatus surface of Claim 24, wherein byproducts generated during an etch process or a deposition process adhere to said electrochemically roughened surface.

32. The semiconductor processing apparatus surface of Claim 31, wherein fluorine and carbon from an etch process react to form a polymer which adheres to said surface.

33. The semiconductor processing apparatus surface of Claim 24, wherein said surface is present on an apparatus component selected from the group consisting of: a wall liner, a cathode liner, a slit valve door, a slit valve liner, a buffer insert, and a gas distribution plate.

34. A method for electrochemically roughening a surface comprising aluminum or an aluminum alloy, including the steps of:

a) immersing said surface in an HCl solution having a concentration ranging from about 1 volume % to about 5 volume %, at a temperature ranging from about 45°C to about 80°C; and

b) applying an electrical charge having a charge density ranging from about

80 amps/ft.^{2} to about 250 amps/ft.^{2} for a time period ranging from about 4 minutes to about 25 minutes.

35. The method of Claim 34, wherein said HCl solution has a concentration ranging from about 1 volume % to about 3 volume %.

36. The method of Claim 35, wherein said temperature of said HCl solution ranges from about 50°C to about 70°C.

37. The method of Claim 34, wherein said HCl solution further includes a chelating agent, and wherein said chelating agent is present at a concentration within the range of about

0.5 volume % to about 3 volume %.

38. The method of Claim 37, wherein said chelating agent is gluconic acid.

39. The method of Claim 34, wherein said charge density ranges from about 120 amps/ft.^{2} to about 250 amps/ft.^{2}.

40. The method of Claim 34, wherein said time period ranges from about 4 minutes to about 20 minutes.

41. The method of Claim 34, wherein said aluminum-comprising surface is an aluminum alloy selected from the group consisting of 6061 and LP.

42. The method of Claim 41, wherein said HCl solution concentration ranges from about 1 volume % to about 1.5 volume %; wherein said temperature of said HCl solution ranges from about 55°C to about 65°C; and wherein said charge density ranges from about 175 amps/ft.^{2} to about 250 amps/ft.^{2}.

43. The method of Claim 42, wherein said HCl solution further includes a gluconic acid chelating agent, which is present at a concentration within the range of about
0.9 volume % to about 1.1 volume %.

44. The method of Claim 43, wherein said time period during which said charge density is present ranges from about 6 minutes to about 12 minutes, and the aluminum alloy is 6061.

45. The method of Claim 43, wherein said wherein said time period during which said charge density is present ranges from about 4 minutes to about 8 minutes, and the aluminum alloy is LP.

Fig. 1

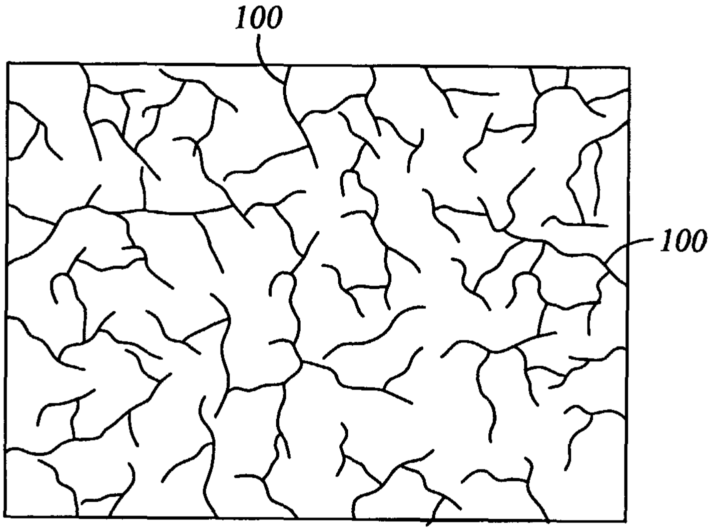


Fig. 2

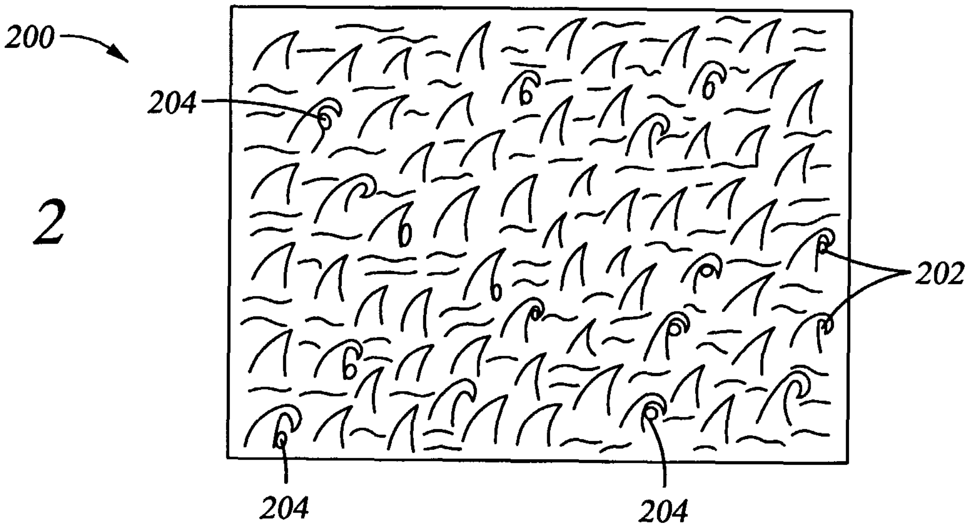
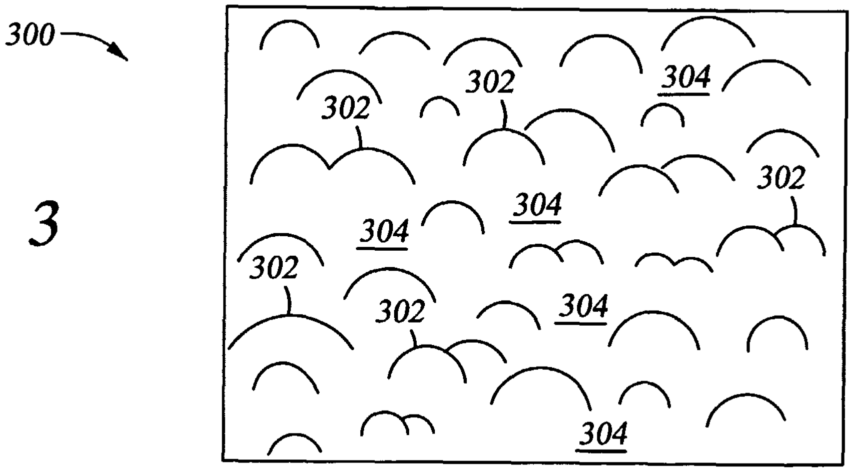


Fig. 3



INTERNATIONAL SEARCH REPORT

Inte al Application No
PCT/US 02/23287

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C23C16/44 C23C16/458 C25F3/04

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)
IPC 7 C23C C25F C25D

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 practical, search terms used)

EPO-Internal, PAJ, 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 6 063 203 A (SATOH KIYOSHI) 16 May 2000 (2000-05-16)	1,4,5,7-9,18-20,24,27,28,30,31
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A	US 3 963 594 A (BRASKO PETER) 15 June 1976 (1976-06-15) claims	34-45

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search 21 November 2002	Date of mailing of the international search report 28/11/2002
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Authorized officer Patterson, A

INTERNATIONAL SEARCH REPORT

Information on patent family members

Inte nal Application No
PCT/US 02/23287

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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