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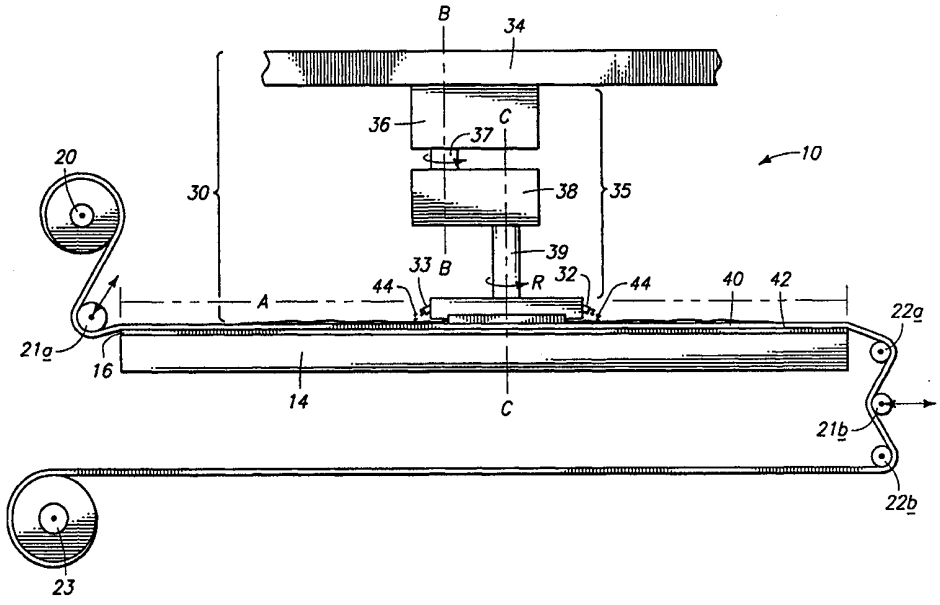
(54) Title: TUNGSTEN CHEMICAL-MECHANICAL POLISHING PROCESS USING A FIXED ABRASIVE POLISHING PAD AND A TUNGSTEN LAYER CHEMICAL-MECHANICAL POLISHING SOLUTION SPECIFICALLY ADAPTED FOR CHEMICAL-MECHANICAL POLISHING WITH A FIXED ABRASIVE PAD

(57) Abstract

The invention comprises tungsten chemical-mechanical polishing processes using fixed abrasive polishing pads, and to tungsten layer chemical-mechanical polishing solutions specifically adapted for chemical-mechanical polishing with fixed abrasive pads. In one implementation, a semiconductor wafer having a layer comprising tungsten at greater than or equal to 50 % molar is provided. Such is positioned in proximity with a fixed abrasive chemical-mechanical polishing pad. A tungsten layer chemical-mechanical polishing solution is provides intermediate the wafer and pad. The solution comprises a tungsten oxidizing component present at form about 0.5 % to 15 % by volume and a pH of less than or equal to about 6.0. The tungsten comprising layer is chemical-mechanical polished with the fixed abrasive pad with the tungsten layer chemical-mechanical polishing solution being received between the wafer and the pad. In one implementation, tungsten from the layer is oxidized with a solution comprising a tungsten oxidizing component present at from about 0.5 % to 15 % by volume and a pH of less than or equal to about 6.0. One or both of tungsten and tungsten oxide is then polished from the tungsten comprising layer with a fixed abrasive chemical-mechanical polishing pad. In one implementation, the invention comprises a tungsten layer chemical-mechanical polishing solution.



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# DESCRIPTION

# TUNGSTEN CHEMICAL-MECHANICAL POLISHING PROCESS USING A FIXED ABRASIVE POLISHING PAD AND A TUNGSTEN LAYER CHEMICAL-MECHANICAL POLISHING SOLUTION SPECIFICALLY ADAPTED FOR CHEMICAL-MECHANICAL POLISHING WITH A FIXED ABRASIVE PAD

## Technical Field

This invention relates to tungsten chemical-mechanical polishing processes using fixed abrasive polishing pads, and to tungsten layer chemical-mechanical polishing solutions specifically adapted for chemical-mechanical polishing with fixed abrasive pads.

## Background Art

In the manufacture of integrated circuits from semiconductor wafers and substrates, wafer polishing is a common technique utilized to remove material and/or achieve planarity. Such polishing can be conducted by purely chemical, purely mechanical or chemical-mechanical polishing means (CMP). With CMP, polishing and removal occurs by a combination of both chemical and mechanical polishing action. CMP utilizes a combination of solid abrasives and chemicals to achieve the combination polishing action. One type of chemical-mechanical polishing utilizes a slurry comprising very hard, solid abrasive particles suspended in a chemical solution. The slurry is interposed between a pad and a wafer, with both typically being caused to rotate, and material removed from the wafer by both chemical and mechanical action. Another form of CMP provides abrasive material embedded within the surface of the polishing pad, and is commonly referred to as fixed abrasive CMP.

Unfortunately, conventional CMP slurries designed for non-fixed abrasive CMP create problems and do not always work satisfactorily in fixed abrasive CMP processes. This has been discovered to be particularly true in the CMP of layers where tungsten is present at greater than or equal to fifty atomic percent. Tungsten finds existing use as a contact/plugging material in fabrication of DRAM and other circuitry. While CMP of tungsten has been reported using non-fixed abrasive pads and slurries, existing materials have proven less than satisfactory when using fixed abrasive pads. Accordingly, needs remain for improved chemical-mechanical processes using fixed abrasive pads and in the development of polishing solutions therefor.

**Brief Description of the Drawings**

Fig. 1 is a diagrammatic view of a prior art chemical-mechanical polishing apparatus used in accordance with an aspect of the invention.

**Best Modes for Carrying Out the Invention and Disclosure of Invention**

The invention comprises tungsten chemical-mechanical polishing processes using fixed abrasive polishing pads, and to tungsten layer chemical-mechanical polishing solutions specifically adapted for chemical-mechanical polishing with fixed abrasive pads. In the context of this document unless otherwise specifically literally narrowed, a "tungsten layer" constitutes a layer having tungsten present at least at 50% molar. In one implementation, a semiconductor wafer having a layer comprising tungsten at greater than or equal to 50% molar is provided. Such is positioned in proximity with a fixed abrasive chemical-mechanical polishing pad. A tungsten layer chemical-mechanical polishing solution is provided intermediate the wafer and pad. The solution comprises a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0. The tungsten comprising layer is chemical-mechanical polished with the fixed abrasive pad with the tungsten layer chemical-mechanical polishing solution being received between the wafer and the pad. Believed process mechanisms by which polishings occur in accordance with the invention are disclosed, but are not intended to be limiting unless expressly worded in the respective claim. In one implementation, the invention comprises a tungsten layer chemical-mechanical polishing solution.

In accordance with one aspect of the invention, a semiconductor wafer having an outer layer or region comprising tungsten at greater than or equal to 50% molar is polished with improved solutions using fixed abrasive pads. The invention was principally predicated upon the chemical-mechanical polishing of tungsten layers consisting essentially of elemental tungsten. However, the invention is anticipated to have applicability to polishing any tungsten layers having greater than or equal to 50% molar tungsten content, including such layers consisting essentially of tungsten alloys.

A wafer having such a layer is positioned within a suitable chemical-mechanical polishing apparatus comprising a fixed abrasive chemical-mechanical polishing pad. Any suitable apparatus is usable, such as by way of example only, those apparatus which rotate/revolve both a fixed abrasive pad and the wafer, those apparatus which rotate/move a fixed abrasive pad and not the wafer, those apparatus which rotate/revolve the wafer and not a fixed

abrasive pad, etc. Fixed abrasive pads can be purchased from many suppliers, such as the 3M Company of St. Paul, Minnesota, U.S.A. A tungsten layer chemical-mechanical polishing solution in accordance with the invention is provided intermediate the pad and the wafer utilizing the polishing equipment. In one implementation, the solution comprises a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0.

Example preferred oxidizing components include nitrate and other compounds. Specific examples include potassium permanganate, ferric nitrate, hydrogen peroxide, nitric acid, ammonium persulfate, potassium iodate, potassium nitrate, ammonium molybdate and mixtures thereof. Solution pH is more preferably kept at less than 4.0, such as from 3.0 to 4.0. The solution also preferably comprises a pH buffer, with potassium hydrogen phthalate, ammonium citrate, ammonium phosphate and mixtures thereof being examples. A preferred concentration range for the buffer is from about 0.1% to 10% by weight. The solution is most ideally provided to be void of solid abrasive material, at least prior to starting chemical-mechanical polishing. Once polishing commences with the fixed abrasive pad, it is possible that some abrasive material from the pad will break off of the pad and find its way into solution, but ideally the solution is essentially abrasive-free in accordance with the most preferred aspects of the invention. The solution can also comprise a surfactant, for example present at a concentration of from 1% to 10% by volume. Example surfactants include polyethylene glycol, polyoxyethylene ether, glycerol, polypropylene glycol, polyoxyethylene lauryl ether, polyoxyethylene cetyl ether, polyoxyethylene stearyl ether, polyoxyethylene oleyl ether, and mixtures thereof. The solution might further comprise thickeners to achieve desired viscosity, with an example preferred range being from about 10 centipoise to 20 centipoise at room temperature. Examples of thickeners include Polyox™ available from Union Carbide of Danbury, Connecticut, U.S.A., and Carbopol™ available from B.F. Goodrich of Cleveland, Ohio, U.S.A.

The solution also preferably comprises a complexing agent in solution for complexing tungsten or an oxidation product thereof. Preferred complexants are amine compounds. Specific examples are triethanolamine, triethylenediamine, ammonium citrate, ammonium phosphate, ammonium oxalate, ammonium carbonate and mixtures thereof. A preferred concentration range for the complexing agent is from about 0.1% to about 10% by weight.

The solution also preferably comprises a tungsten corrosion inhibitor (i.e., preferably at a concentration of from 0.01% to 2% by weight) to protect tungsten in the low lying areas and to improve planarity and dishing performance of the polish. Examples include phosphates, polyphosphates and silicates, with potassium hypophosphite and potassium silicate being but two specific examples.

With such exemplary solutions in accordance with the invention received intermediate the wafer and pad, the tungsten comprising layer is chemical-mechanical polished with the fixed abrasive pad.

In another considered aspect of the invention, tungsten from the layer being polished is oxidized with a solution comprising a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0. One or both of tungsten and tungsten oxide from the tungsten comprising layer is polished with a fixed abrasive chemical-mechanical polishing pad. The oxidized tungsten might oxidize on the layer prior to removal, or be polished away from the layer as tungsten and be oxidized and dissolved in solution thereafter. Both of these actions might also occur, with one or the other possibly predominating. Solution components during polishing preferably constitute those as described above, and again in significant consideration is ideally void of solid abrasive material.

Polishing of tungsten is preferably conducted with soft abrasives, such as $SiO_2$, to reduce or avoid scratch or chatter marks from occurring on the polished tungsten surface. Further, pH is preferably kept below 4 to facilitate the polish. Yet in prior art chemical-mechanical polishing solutions and processes, it has been found that $SiO_2$ abrasives do not remain well dispersed throughout the solution when pH falls below 4.0. This has typically resulted in using additional or other harder abrasive materials than desired to achieve a suitable solution during polish. Fixed abrasive polishing using solutions of the invention can enable utilization of $SiO_2$ as the abrasive on the pad in combination with pH's at or below 4.0.

Further in many instances, it would be desirable to maintain temperature at or below room temperature during the tungsten polish (i.e., at or below 74°F). This is seldom practical where lower slurry temperature will also result in poor abrasive material dispersal throughout the slurry during polish. Accordingly, elevated temperatures are utilized during the polish. Yet in accordance with an aspect of the invention, slurry temperature during polish can be maintained at or below room temperature (further preferably at or below

65°F) where the abrasive is fixed on the pad and agglomeration of abrasives within the slurry is not a concern. Polishing in all embodiments preferably is conducted at atmospheric pressure and anywhere from 40°F up to 145°F, although other conditions are of course possible.

Example equipment and processing utilized in accordance with the invention is described with reference to Fig. 1, wherein a web-format planarizing machine 10 is used for chemical-mechanical polishing a semiconductor wafer 12. Planarizing machine 10 has a support table 14 with a top panel 16 at a work station where an operative portion (A) of a planarizing pad 40 is positioned. The top-half panel 16 is generally a rigid plate to provide a flat, solid surface to which a particular section of planarizing pad 40 may be secured during polishing.

Planarizing machine 10 also has a plurality of rollers to guide, position and hold planarizing pad 40 over top panel 16. The rollers include a supply roller 20, first and second idler rollers 21a and 21b, first and second guide rollers 22a and 22b, and a take-up roller 23. The supply roller 20 carries an unused or pre-operative portion of the planarizing pad 40, and take-up roller carries a used or post-operative portion of planarizing pad 40. First idler roller 21a and first guide roller 22a stretch planarizing pad 40 over top panel 16 to hold the planarizing pad 40 stationary during operation. Planarizing pad 40 in accordance with the invention preferably comprises a fixed abrasive pad, such as described above, and having a length greater than a maximum diameter of the wafers being polished. A motor (not shown) drives at least one of supply roller 20 and take-up roller 23 to sequentially advance the planarizing pad 40 across the top-panel 16. As such, clean pre-operative sections of the planarizing pad 40 may be quickly substituted for used sections to provide a consistent surface for planarizing and/or cleaning the substrate.

The web-format planarizing machine 10 has a carrier assembly 30 that controls and protects wafer 12 during polishing. Carrier assembly 30 generally has a substrate holder 32 to pick up, hold and release wafer 12 at appropriate stages of the polishing cycle. A plurality of nozzles 33 attached to substrate holder 32 dispense a planarizing solution 44 in accordance with the invention onto a planarizing surface 42 of planarizing pad 40. Carrier assembly 30 also generally has a support gantry 34 carrying a drive assembly 35 that translates along gantry 34. Drive assembly 35 generally has an actuator 36, a drive shaft 37 coupled to the actuator 36, and an arm 38 projecting from drive

shaft 37. Arm 38 carries substrate holder 32 via another shaft 39 such that drive assembly 35 orbits substrate holder 32 about an axis B-B offset from a center point C-C of wafer 12.

In accordance with an aspect of the invention, the process preferably comprises moving web/pad 40 a distance less than the maximum diameter of the wafer such that a subsequently polished wafer is exposed to a fresh pad segment which was not utilized to polish the immediately preceding wafer. Preferably, the distance moved is less than or equal to 1.0% of the maximum diameter for uniformity of polish and to extend life of the pad. For example for an 8-inch diameter wafer, an incremental movement of pad/web 40 between each polishing is 0.25 inch.

## CLAIMS

1. A chemical-mechanical polishing process comprising:

providing a semiconductor wafer having a layer comprising tungsten at greater than or equal to 50% molar;

positioning the wafer in proximity with a fixed abrasive chemical-mechanical polishing pad;

providing a tungsten layer chemical-mechanical polishing solution intermediate the wafer and pad, the tungsten layer chemical-mechanical polishing solution comprising a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0; and

chemical-mechanical polishing the tungsten comprising layer with the fixed abrasive pad with the tungsten layer chemical-mechanical polishing solution being received between the wafer and the pad.

2. The chemical-mechanical polishing process of claim 1 wherein the solution pH is 4.0 or less.

3. The chemical-mechanical polishing process of claim 1 wherein the solution pH is from 3.0 to 4.0.

4. The chemical-mechanical polishing process of claim 1 comprising complexing tungsten or an oxidation product thereof with a complexing agent in the solution comprising from 0.1% to 10% by weight of the solution.

5. The chemical-mechanical polishing process of claim 4 wherein the complexing agent is selected from the group consisting of triethanolamine, trietheylenediamine, ammonium citrate, ammonium phosphate, ammonium oxalate, ammonium carbonate and mixtures thereof.

6. The chemical-mechanical polishing process of claim 1 wherein the solution is void of solid abrasive material at least prior to said chemical-mechanical polishing.

7. The chemical-mechanical polishing process of claim 1 wherein the layer consists essentially of elemental tungsten.

8. The chemical-mechanical polishing process of claim 1 wherein the layer consists essentially of a tungsten alloy.

9. The chemical-mechanical polishing process of claim 1 wherein the oxidizing component comprises a nitrate compound.

10. The chemical-mechanical polishing process of claim 1 wherein the oxidizing component is selected from the group consisting of potassium permanganate, ferric nitrate, hydrogen peroxide, nitric acid, ammonium persulfate, potassium iodate, potassium nitrate, ammonium molybdate and mixtures thereof.

11. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a surfactant present at a concentration of from 1% to 10% by volume.

12. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a surfactant selected from group consisting of polyethylene glycol, polyoxyethylene ether, glycerol, polypropylene glycol, polyoxyethylene lauryl ether, polyoxyethylene cetyl ether, polyoxyethylene stearyl ether, polyoxyethylene oleyl ether, and mixtures thereof.

13. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a thickener and has a viscosity of from about 10 centipoise to 20 centipoise at room temperature.

14. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a tungsten corrosion inhibitor comprising from 0.01% to 2% by weight of the solution.

15. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a tungsten corrosion inhibitor selected from the group consisting of phosphates, polyphosphates, silicates and mixtures thereof.

16. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a pH buffer comprising from 0.1% to 10% by weight of the solution.

17. The chemical-mechanical polishing process of claim 1 wherein the solution comprises a pH buffer selected from the group consisting of potassium hydrogen phthalate, ammonium citrate, ammonium phosphate and mixtures thereof.

18. The chemical-mechanical polishing process of claim 1 wherein a majority of fixed abrasives on the pad comprise $SiO_2$.

19. The chemical-mechanical polishing process of claim 1 wherein a majority of fixed abrasives on the pad comprise $SiO_2$ and the solution pH is less than or equal to 4.0.

20. The chemical-mechanical polishing process of claim 1 comprising maintaining temperature of the solution during polishing at or below about 74°F.

21. The chemical-mechanical polishing process of claim 1 comprising maintaining temperature of the solution during polishing at or below about 65°F.

22. The chemical-mechanical polishing process of claim 1 further comprising chemical-mechanical polishing another of said semiconductor wafer in another polishing step using the fixed abrasive pad, the fixed abrasive pad comprising an elongated web of fixed abrasive pad material having a length greater than a maximum diameter of the said wafers being polished, the process comprising moving the web a distance less than the maximum diameter to polish the another wafer with a fresh pad segment not utilized to polish the first polished wafer.

23. The chemical-mechanical polishing process of claim 22 wherein the distance is less than or equal to 1.0% of the maximum diameter.

24. A chemical-mechanical polishing process comprising:

providing a semiconductor wafer having a layer comprising tungsten at greater than or equal to 50% molar;

oxidizing tungsten from the layer with a solution comprising a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0; and

polishing one or both of tungsten and tungsten oxide from the tungsten comprising layer with a fixed abrasive chemical-mechanical polishing pad.

25. The chemical-mechanical polishing process of claim 24 wherein the tungsten oxidizing predominately occurs before removal from the layer.

26. The chemical-mechanical polishing process of claim 24 wherein the tungsten oxidizing predominately occurs after removal from the layer.

27. The chemical-mechanical polishing process of claim 24 wherein the tungsten oxidizing occurs both before and after removal from the layer.

28. The chemical-mechanical polishing process of claim 24 wherein the solution is void of solid abrasive material at least prior to said polishing.

29. The chemical-mechanical polishing process of claim 24 wherein the solution pH is 4.0 or less.

30. The chemical-mechanical polishing process of claim 24 wherein the solution pH is from 3.0 to 4.0.

31. The chemical-mechanical polishing process of claim 24 wherein the solution comprises a pH buffer comprising from 0.1% to 10% by weight of the solution.

32. The chemical-mechanical polishing process of claim 24 wherein the solution comprises a pH of from 3.0 to 4.0, and comprises a pH buffer comprising from 0.1% to 10% by weight of the solution.

33. The chemical-mechanical polishing process of claim 24 wherein the solution comprises a pH buffer selected from the group consisting of potassium hydrogen phthalate, ammonium citrate, ammonium phosphate and mixtures thereof.

34. The chemical-mechanical polishing process of claim 24 wherein the layer consists essentially of elemental tungsten.

35. The chemical-mechanical polishing process of claim 24 wherein the layer consists essentially of a tungsten alloy.

36. The chemical-mechanical polishing process of claim 24 comprising complexing oxidized tungsten with a complexing agent in the solution comprising from 0.1% to 10% by weight of the solution.

37. The chemical-mechanical polishing process of claim 36 wherein the complexing agent is selected from the group consisting of triethanolamine, trietheylenediamine, ammonium citrate, ammonium phosphate, ammonium oxalate, ammonium carbonate and mixtures thereof.

38. The chemical-mechanical polishing process of claim 24 wherein the solution comprises a tungsten corrosion inhibitor comprising from 0.01% to 2% by weight of the solution.

39. The chemical-mechanical polishing process of claim 24 wherein the solution comprises a tungsten corrosion inhibitor selected from the group consisting of phosphates, polyphosphates, silicates and mixtures thereof.

40. The chemical-mechanical polishing process of claim 24 wherein a majority of fixed abrasives on the pad comprise $SiO_2$.

41. The chemical-mechanical polishing process of claim 24 wherein a majority of fixed abrasives on the pad comprise $SiO_2$ and the solution pH is less than or equal to 4.0.

42. The chemical-mechanical polishing process of claim 24 comprising maintaining temperature of the solution during polishing at or below about 74°F.

43. The chemical-mechanical polishing process of claim 24 comprising maintaining temperature of the solution during polishing at or below about 65°F.

44. The chemical-mechanical polishing process of claim 24 further comprising chemical-mechanical polishing another of said semiconductor wafer in another polishing step using the fixed abrasive pad, the fixed abrasive pad comprising an elongated web of fixed abrasive pad material having a length greater than a maximum diameter of the said wafers being polished, the process comprising moving the web a distance less than the maximum diameter to polish the another wafer with a fresh pad segment not utilized to polish the first polished wafer.

45. The chemical-mechanical polishing process of claim 44 wherein the distance is less than or equal to 1.0% of the maximum diameter.

46. A tungsten layer chemical-mechanical polishing solution specifically adapted for chemical-mechanical polishing with a fixed abrasive pad, the solution comprising a tungsten oxidizing component present at from about 0.5% to 15% by volume and a pH of less than or equal to about 6.0.

47. The chemical-mechanical polishing solution of claim 46 and being void of solid abrasive material.

48. The chemical-mechanical polishing solution of claim 46 having a pH of 4.0 or less.

49. The chemical-mechanical polishing solution of claim 46 having a pH from 3.0 to 4.0.

50. The chemical-mechanical polishing solution of claim 46 wherein the oxidizing component comprises a nitrate compound.

51. The chemical-mechanical polishing solution of claim 46 wherein the oxidizing component is selected from the group consisting of potassium permanganate, ferric nitrate, hydrogen peroxide, nitric acid, ammonium persulfate, potassium iodate, potassium nitrate, ammonium molybdate and mixtures thereof.

52. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a surfactant present at a concentration of from 1% to 10% by volume.

53. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a surfactant selected from group consisting of polyethylene glycol, polyoxyethylene ether, glycerol, polypropylene glycol, polyoxyethylene lauryl ether, polyoxyethylene cetyl ether, polyoxyethylene stearyl ether, polyoxyethylene oleyl ether, and mixtures thereof.

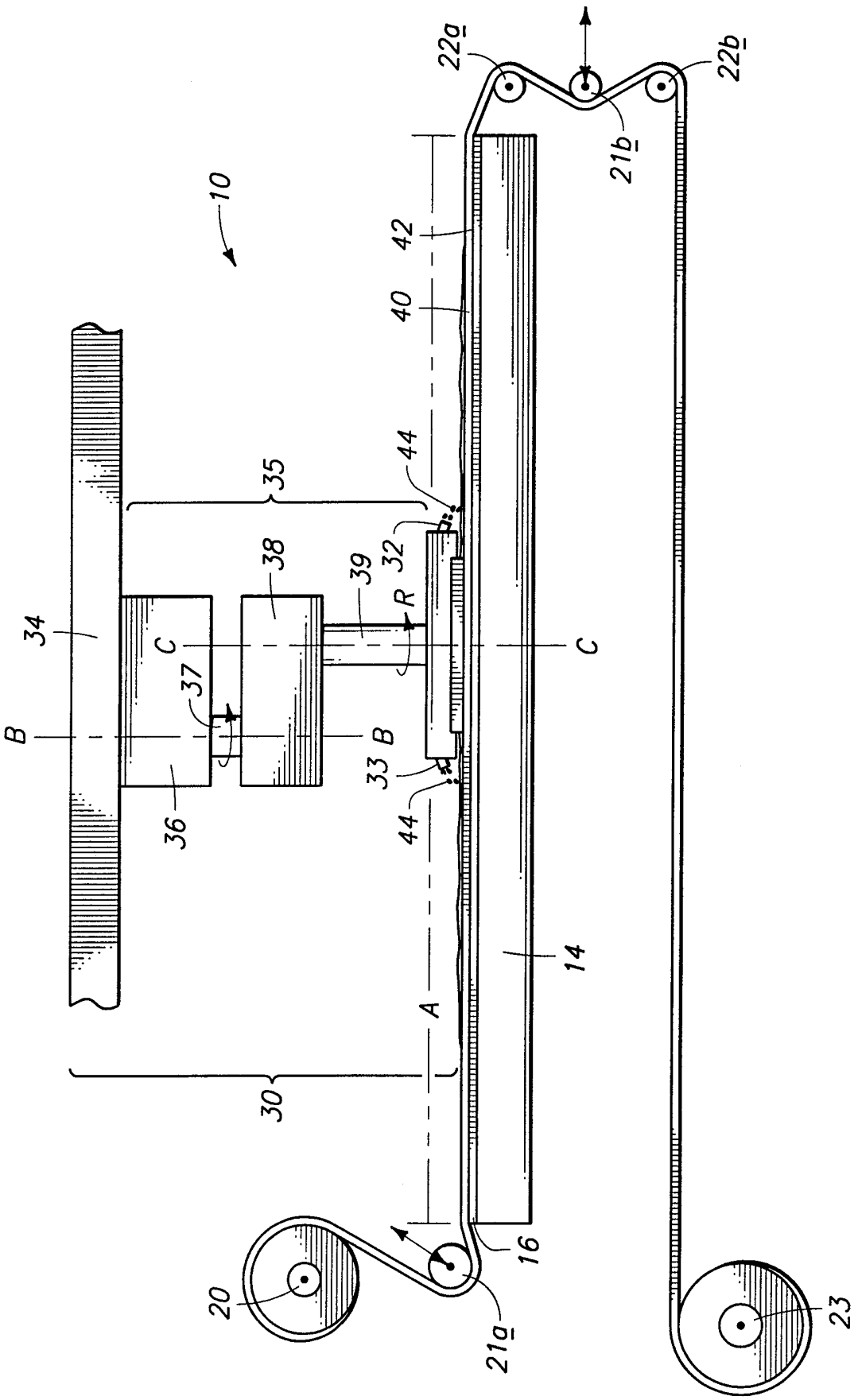
54. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a thickener and has a viscosity of from about 10 centipoise to 20 centipoise at room temperature.

55. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a tungsten corrosion inhibitor comprising from 0.01% to 2% by weight of the solution.

56. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a tungsten corrosion inhibitor selected from the group consisting of phosphates, polyphosphates, silicates and mixtures thereof.

57. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a pH buffer comprising from 0.1% to 10% by weight of the solution.

58. The chemical-mechanical polishing solution of claim 46 wherein the solution comprises a pH buffer selected from the group consisting of potassium hydrogen phthalate, ammonium citrate, ammonium phosphate and mixtures thereof.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# INTERNATIONAL SEARCH REPORT

Inter. onal Application No
PCT/US 99/27033

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01L21/321 C09G1/02 C09K3/14

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 H01L C09G C09K

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)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category ° | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| X | WO 98 18159 A (MICRON TECHNOLOGY)<br>30 April 1998 (1998-04-30)<br><br>the whole document | 1,6,7,9,<br>10,18,<br>24-28,<br>34,40,<br>46,47,<br>50,51 |
| Y | --- | 1-58 |
| Y | WO 98 49723 A (MINNESOTA MINING AND MANUFACTURING)<br>5 November 1998 (1998-11-05)<br>claims 8,13 | 1-58 |
| A | --- | 2,3,16,<br>29-31,57 |
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