Claims

1. A method of evaluating whether additives in an electroplating bath of interest meet an electroplating specification, the method comprising:

performing a first experiment by:

contacting an electrode with the electroplating bath of interest,

applying a current density waveform to the electrode, wherein the current density waveform applied approximates a current density experienced in a field region of a substrate when electroplated in the electroplating bath of interest, and

recording a first potential trace output during the first experiment;

performing a second experiment by:

contacting a second electrode with an acceleration solution comprising accelerator until the second electrode is substantially fully accelerated,

rinsing the acceleration solution from the second electrode,

contacting the second electrode with the electroplating bath of interest,

applying a second current density waveform or a potential waveform to the second electrode, wherein the second current density waveform or potential waveform approximates the current density or potential experienced within a feature on the substrate when electroplated in the electroplating bath of interest,

where a second current density waveform is applied, recording a second potential trace output during the second experiment, and where a potential waveform is applied to the second electrode, recording a current trace output during the second experiment; and

determining, based on two or more parameters selected from the group consisting of the first potential trace output, the second potential trace output, the current trace output, and calibration

data, whether the additives in the electroplating bath of interest meet the electroplating specification.

2. The method of claim 1, wherein the electroplating specification relates to the capability of the additives in the electroplating bath of interest to fully fill the feature on the substrate through a bottom-up mechanism in an acceptable timeframe.

3. The method of claim 1, wherein the current density waveform applied during the first experiment corresponds to a current density waveform used for electroplating material on the substrate in a target filling process.

4. The method of claim 3, wherein the current density waveform applied during the first experiment corresponds to a current density waveform used for electroplating material on the substrate in a target filling process, as modified by a field current density correction factor.

5. The method of claim 3, wherein a rotation rate of the second electrode during the second experiment is higher than a rotation rate of the ~~semiconductor~~ substrate during the target filling process.

6. The method of claim 3, wherein a temperature of the electroplating bath of interest during the second experiment is higher than a temperature of the electroplating bath of interest during the target filling process.

7. The method of claim 1, wherein the first potential trace output from the first experiment is used to select the potential waveform applied in the second experiment.

8. The method of claim 7, wherein the potential waveform is selected based on the potential experienced during a final period of electroplating during the first experiment.

9. The method of claim 8, wherein the potential waveform is selected based on an average potential of the first potential trace output during a final period of electroplating during the first experiment.

10. The method of claim 7, wherein the first potential trace output from the first experiment is used to calculate a predicted potential, the predicted potential corresponding to a potential that would be experienced if electroplating were to continue after a final electroplating period in the first experiment, and wherein the potential waveform in the second experiment is based on the predicted potential.

11. The method of claim 2, wherein the second current density waveform is selected based on a current density to fully fill the feature in the acceptable timeframe.

12. The method of claim 1, wherein the electrode and the second electrode are the same electrode, and further comprising removing material deposited on the electrode during the first experiment before contacting the electrode with the acceleration solution.

13. The method of claim 1, wherein the determining comprises comparing a current density at a relevant time from the current density trace output from the second experiment to a threshold current density.

14. The method of claim 1, wherein the determining comprises comparing a time at which the current density trace output from the second experiment reaches a threshold current density to a threshold time.

15. The method of claim 1, wherein the determining comprises integrating the current density trace output from the second experiment between a first time and a second time to calculate a charge density, and comparing the charge density to a threshold charge density.

16. The method of claim 1, wherein the determining comprises calculating a ratio between a potential from the first potential trace output at a first time during the first experiment to a potential from the second potential trace output at a second time during the second experiment, and comparing the ratio to a threshold ratio.

17. The method of claim 1, wherein the calibration data is generated by performing the first and/or second experiment on electrolytes that are known to produce acceptable fill results and electrolytes that are known to produce unacceptable fill results.

18. The method of claim 17, wherein the calibration data comprises one or more of the parameters selected from the group consisting of: a threshold current density, a threshold charge density, a threshold time, and a threshold ratio of potentials.

19. The method of claim 2, wherein the acceptable timeframe is about 1 hour or less.

20. The method of claim 1, further comprising electroplating metal onto the electrode before contacting the electrode with the electroplating bath of interest in the first experiment, and electroplating metal onto the second electrode before contacting the second electrode with the acceleration solution.

21. The method of claim 1, wherein a concentration of accelerator in the acceleration solution is at least ~~about~~ 10 times a concentration of accelerator in the electroplating bath of interest.

22. A method of monitoring the additives in an electroplating bath of interest, the method comprising:

(a) applying a defined current density to a first electrode while in contact with the electroplating bath of interest,

wherein the defined current density represents a current density experienced in a field region of a substrate when electroplated in the electroplating bath of interest, and

wherein the first electrode's surface is not substantially fully accelerated;

(b) recording a potential trace output of the first electrode when applying the defined current density;

(c) applying a second defined current density or a defined potential to a substantially fully accelerated surface of a second electrode while in contact with the electroplating bath of interest,

wherein the second defined current density or the defined potential represents the current density or potential experienced within a feature on the substrate when electroplated in the electroplating bath of interest;

(d) recording a second potential trace output and/or a current density trace output of the second electrode when applying the second defined current density or the defined potential; and

(e) determining, based on information contained in one or more of the outputs, whether the additives of the electroplating bath of interest meet a defined electroplating specification.

23. An apparatus for evaluating whether additives in an electroplating bath of interest meet an electroplating specification, the apparatus comprising:

an analysis chamber;

one or more inlets for providing to the analysis chamber a test bath solution from an electroplating bath of interest and one or more additional solutions;

an outlet for removing fluid from the analysis chamber;

a working electrode;

a power supply; and

a controller ~~having instructions~~configured to cause:

(a) performing a first experiment while the test bath solution is present in the analysis chamber by applying a defined current density to the working electrode, and recording a potential trace output of the working electrode;

(b) contacting the working electrode with an acceleration solution for a duration sufficient to substantially fully saturate the working electrode with accelerator;

(c) performing a second experiment while test bath solution is present in the analysis chamber by either (i) applying a second defined current density to the working electrode and recording a second potential trace output of the working electrode, or (ii) applying a defined potential to the working electrode and recording a current density trace output; and

(d) determininge, based on information contained in one or more of the potential trace output, the second potential trace output, the current density trace output, and calibration data, whether the electroplating additives in the electroplating bath of interest meet the electroplating specification.

24. The apparatus of claim 23, wherein the controller is further comprises instructions forconfigured to cause:

prior to (a):

flowing a standardized electroplating solution into the analysis chamber and electroplating metal onto the working electrode while the working electrode is in contact with the standardized electroplating solution,

removing the standardized electroplating solution from the analysis chamber, and

flowing the test bath solution into the analysis chamber.

25. The apparatus of claim 24, wherein the controller is further comprises instructions forconfigured to:

after (a) and before (b):

removing material electroplated onto the working electrode,

removing the test bath solution from the analysis chamber; and

after (b) and before (c):

rinsing the working electrode to thereby remove unadsorbed acceleration solution, and

flowing test bath solution into the analysis chamber.

26. The apparatus of claim 25, wherein the ~~instructions for~~controller is configured to cause removing material electroplated onto the working electrode ~~comprise instructions to apply a~~by causing a reverse current to be applied to the working electrode to thereby deplate the material.

27. The apparatus of claim 25, wherein the ~~instructions for~~controller is configured to cause removing material electroplated onto the working electrode ~~comprise instructions to flow~~ by causing a chemical etching solution to flow into the analysis chamber to thereby chemically etch the material from the working electrode.

28. The apparatus of claim 23, wherein the ~~instructions in (b) further comprise instructions for~~controller is configured to cause flowing the acceleration solution into the analysis chamber in (b) to thereby contact the working electrode with the acceleration solution, and ~~further comprising instructions for~~to cause removing the acceleration solution from the analysis chamber and rinsing the analysis chamber with a rinse solution prior to (c).

29. The apparatus of claim 23, wherein the electroplating specification relates to a capability of the additives in the electroplating bath of interest to fully fill a feature on a substrate through a bottom-up mechanism in an acceptable timeframe.

30. The apparatus of claim 23, wherein the ~~instructions in~~controller is configured to cause (d) ~~comprise instructions to compare~~by comparing a current density at a relevant time from the current density trace output from (c) to a threshold current density.

31. The apparatus of claim 23, wherein the ~~instructions in~~controller is configured to cause (d) ~~comprise instructions to compare~~by comparing a time at which the current density trace output from (c) reaches a threshold current density to a threshold time.

32. The apparatus of claim 23, wherein the ~~instructions in~~controller is configured to cause (d) ~~comprise instructions to integrate~~by integrating the current density trace output from (c) between a first time and a second time to calculate a charge density, and comparing the charge density to a threshold charge density.

33. The apparatus of claim 23, wherein the ~~instructions in~~controller is configured to cause (d) ~~comprise instructions to calculate~~by calculating a ratio between a potential from the first potential trace output at a first time in (a) to a potential from the second potential trace output at a second time in (c), and comparing the ratio to a threshold ratio.

34. The apparatus of claim 23, wherein the calibration data comprises one or more of the parameters selected from the group consisting of: a threshold current density, a threshold charge density, a threshold time, and a threshold ratio of potentials.

35. The apparatus of claim 23, further comprising a counter electrode and a membrane separating the counter electrode from the working electrode.

36. The apparatus of claim 23, wherein the working electrode is a rotating disk electrode.

37. The apparatus of claim 23, further comprising a channel through which fluid may flow, wherein the working electrode is positioned in/on the channel.

38. The apparatus of claim 23, wherein at least one of the one or more inlets are connected with an electroplating apparatus for electroplating metal onto semiconductor substrates.

39. The apparatus of claim 23, further comprising a gas inlet for providing inert gas to the analysis chamber.

40. The apparatus of claim 23, wherein the controller ~~further comprises instructions for~~is configured to cause applying vacuum conditions to the analysis chamber.

41. A method of analyzing an electroplating bath of interest, the method comprising:

(a) applying a current to a first electrode while in contact with the electroplating bath of interest, wherein the first electrode's surface is not substantially fully accelerated;

(b) recording an electrical output of the first electrode produced in response to the applied current;

(c) applying a second current or a potential to a substantially fully accelerated surface of a second electrode while in contact with the electroplating bath of interest,

(d) recording a second electrical output of the second electrode when applying the second current or the potential; and

(e) determining, based on information contained in the outputs, whether the electroplating bath of interest meet a defined electroplating specification.

42. The method of claim 41, wherein the first and second electrodes are the same electrode with different surface metals deposited thereon.

~~43. An apparatus for evaluating whether additives in an electroplating bath of interest meet an electroplating specification, the apparatus comprising:~~

~~an analysis chamber;~~

~~one or more inlets for providing to the analysis chamber a test bath solution from an electroplating bath of interest and one or more additional solutions;~~

~~an outlet for removing fluid from the analysis chamber;~~

~~a working electrode;~~

~~a power supply; and~~

~~a controller configured to cause:~~

~~(a) performing a first experiment while the test bath solution is present in the analysis chamber by applying a defined current density to the working electrode, and recording a potential trace output of the working electrode;~~

~~(b) contacting the working electrode with an acceleration solution for a duration sufficient to substantially fully saturate the working electrode with accelerator;~~

~~(c) performing a second experiment while the test bath solution is present in the analysis chamber by either (i) applying a second defined current density to the working electrode and recording a second potential trace output of the working electrode, or (ii) applying a defined potential to the working electrode and recording a current density trace output; and~~

~~(d) determining, based on information contained in one or more of the potential trace output, the second potential trace output, the current density trace output, and calibration data, whether the additives in the electroplating bath of interest meet the electroplating specification.~~

~~44. The apparatus of claim 43, wherein the defined current density applied to the working electrode in (a) approximates a current density experienced in a field region of a substrate when electroplated in the electroplating bath of interest.~~

~~45. The apparatus of claim 44, wherein the second defined current density or the defined potential applied to the working electrode in (c) approximates a current density or potential, respectively, experienced within a feature on the substrate when electroplated in the electroplating bath of interest.~~

~~46~~43. An apparatus for evaluating whether additives in an electroplating bath of interest meet an electroplating specification, the apparatus comprising:

an analysis chamber;

one or more inlets for providing to the analysis chamber a test bath solution from an electroplating bath of interest and one or more additional solutions;

an outlet for removing fluid from the analysis chamber;

a working electrode;

a power supply; and

a controller configured to cause:

(a) applying a defined current density to the working electrode while in contact with the electroplating bath of interest,

wherein the defined current density represents a current density experienced in a field region of a substrate when electroplated in the electroplating bath of interest, and

wherein the working electrode's surface is not substantially fully accelerated during (a);

(b) recording a potential trace output of the working electrode when applying the defined current density;

(c) applying a second defined current density or a defined potential to a substantially fully accelerated surface of the working electrode while in contact with the electroplating bath of interest,

wherein the second defined current density or the defined potential represents the current density or potential experienced within a feature on the substrate when electroplated in the electroplating bath of interest;

(d) recording a second potential trace output and/or a current density trace output of the working electrode when applying the second defined current density or the defined potential; and

(e) determining, based on information contained in one or more of the potential trace output, the second potential trace output, and the current density trace output, whether the additives of the electroplating bath of interest meet the electroplating specification.

~~47~~44. An apparatus for evaluating whether additives in an electroplating bath of interest meet an electroplating specification, the apparatus comprising:

an analysis chamber;

one or more inlets for providing to the analysis chamber a test bath solution from an electroplating bath of interest and one or more additional solutions;

an outlet for removing fluid from the analysis chamber;

a working electrode;

a power supply; and

a controller configured to cause:

(a) applying a current to the working electrode while in contact with the electroplating bath of interest, wherein the working electrode's surface is not substantially fully accelerated during (a);

(b) recording ~~an~~a first electrical output of the working electrode produced in response to the applied current in (a);

(c) applying a second current or a potential to a substantially fully accelerated surface of the working electrode while in contact with the electroplating bath of interest,

(d) recording a second electrical output of the working electrode when applying the second current or the potential; and

(e) determining, based on information contained in one or more of the first electrical output and the second electrical output, whether the electroplating bath of interest meets the electroplating specification.

~~48~~45. The apparatus of claim ~~47~~44, wherein the current applied to the working electrode in (a) approximates a current density experienced in a field region of a substrate when electroplated in the electroplating bath of interest.

~~49~~46. The apparatus of claim ~~48~~45, wherein the second current or the potential applied to the working electrode in (c) approximates a current density or potential, respectively, experienced within a feature on the substrate when electroplated in the electroplating bath of interest.

~~50~~47. The apparatus of claim ~~47~~44, wherein the controller is configured to cause: prior to (c), contacting the working electrode with an acceleration solution such that the surface of the working electrode becomes substantially fully accelerated.

~~51~~48. The apparatus of claim ~~47~~44, wherein the controller is configured to cause: prior to (a), flowing a standardized electroplating solution into the analysis chamber, electroplating metal onto the working electrode while the working electrode is in contact with the standardized electroplating solution, removing the standardized electroplating solution from the analysis chamber, and flowing the test bath solution into the analysis chamber.

~~52~~49. The apparatus of claim ~~51~~48, wherein the controller is configured to cause: after (b) and before (c), removing metal electroplated onto the working electrode and remov~~eing~~ the test bath solution from the analysis chamber, flowing an acceleration solution into the analysis chamber and contacting the working electrode with the acceleration solution such that the surface of the working electrode becomes substantially fully accelerated, removing the acceleration solution from the analysis chamber, rinsing the working electrode to thereby remove unadsorbed acceleration solution, and flowing the test bath solution into the analysis chamber.

~~53~~50. The apparatus of claim ~~52~~49, wherein the metal electroplated onto the working electrode is removed by (i) applying a reverse current to the working electrode to thereby deplate the metal, or (ii) contacting the working electrode with a chemical etching solution to thereby chemically etch the metal.

~~54~~51. The apparatus of claim ~~47~~44, wherein the electroplating specification relates to a capability of the additives in the electroplating bath of interest to fully fill a feature on a substrate through a bottom-up fill mechanism in an acceptable timeframe.

~~55~~52. The apparatus of claim ~~54~~51, wherein the acceptable timeframe is ~~about~~ 1 hour or less.

~~56~~53. The apparatus of claim ~~47~~44, wherein the controller is configured to cause (e) ~~comprises~~by comparing a current density at a relevant time from the second electrical output to a threshold current density.

~~57~~54. The apparatus of claim ~~47~~44, wherein the controller is configured to cause (e) ~~comprises~~by comparing a time at which a current density from the second electrical output reaches a threshold current density to a threshold time.

~~58~~55. The apparatus of claim ~~47~~44, wherein the controller is configured to cause (e) ~~comprises~~by integrating the second electrical output between a first time and a second time to calculate a charge density, and comparing the charge density to a threshold charge density.

~~59~~56. The apparatus of claim ~~47~~44, wherein the controller is configured to cause (e) ~~comprises~~ by calculating a potential ratio between a first potential from the first electrical output at a first time to a second potential from the second electrical output at a second time, and comparing the potential ratio to a threshold potential ratio.

~~60~~57. The apparatus of claim ~~47~~44, wherein controller is configured to cause (e) ~~comprises~~by comparing information from the first electrical output and/or the second electrical output to a threshold time, a threshold current density, a threshold charge density, and/or a threshold potential ratio.

~~61~~58. The apparatus of claim ~~47~~44, further comprising a counter electrode, a reference electrode, and a membrane separating the counter electrode from the working electrode.

~~62~~59. The apparatus of claim ~~47~~44, wherein the controller is configured to cause applying vacuum conditions to the analysis chamber.